

Honeywell's Request for Updates on the Reexamination Status

16. Since they began, I have been an active participant in the settlement negotiations with counsel for Honeywell in this litigation. The settlement negotiations [REDACTED] [REDACTED] involved an extensive amount of email correspondence as well as occasional joint teleconferences.

17. In connection with the settlement negotiations, Honeywell specifically requested information regarding the reexamination proceedings involving the patents in suit on or before June 19, 2007 when Ms. Huttner (Honeywell's counsel) asked whether a response to the Office Action in the reexamination of US 6,472,128 ('128 reexamination, SN 90/008,359) had been filed. I advised Ms. Huttner that Rohm and Haas had filed a response on June 11, 2007. Ms. Huttner requested me to send her a copy of the response and I did so. Attached hereto as Exhibit M is a true and correct copy of the '128 reexamination response that I sent to Ms. Huttner as an attachment to my letter of June 20, 2007. My June 20, 2007 letter is previously of record as Exhibit D to my first Declaration.

18. Mr. Jacobson requested an update of the reexaminations during the July 2, 2007 teleconference. Rohm and Haas responded that nothing new had occurred. However, Mr. Jacobson (Honeywell's in-house counsel) never requested for an update in writing either prior to or after that teleconference. Mr. Jacobson sent me an email on June 29th prior to the July 2nd teleconference setting forth Honeywell's comments and feedback on the settlement agreement. Attached hereto as Exhibit N is a true and correct copy of Mr. Jacobson's email. Nowhere in this email is there a request for an update on the status of the reexaminations. The "heated" discussion with Mr. Jacobson on the issue of PTO communications involved sending Honeywell copies of documents filed with or received from the PTO that would impact Honeywell's

obligations under the Settlement Agreement. Mr. Jacobson did not mention a concern about being notified as to every aspect of the *ex parte* reexamination that did not involve documents being sent to/from the PTO, such as if and when interviews would occur.

19. Apart from Ms. Huttner's inquiry as to the '128 reexamination status request prior to June 19, 2007 and Mr. Jacobson's oral request mentioned above, neither Ms. Huttner nor Mr. Jacobson specifically requested an update of the status of either the '128 reexamination or the co-pending reexamination of US 6,773,864 ('864 reexamination, SN 90/008,360). I have no evidence that Honeywell ever requested an update on the status of either the '128 or '864 reexaminations. Further, at no time did either Ms. Huttner or Mr. Jacobson send me a letter or email specifically requesting an update for either reexamination, and I note that no such email or letter is attached to Honeywell's Answering Brief on Rohm and Haas's Motion to Enforce the Settlement Agreement or to its supporting papers.

20. Mr. Jacobson did not participate for Honeywell in any settlement teleconferences with Rohm and Haas after July 2, 2007, however he did participate by email correspondence.

21. On July 9th, I became aware that Rohm and Haas's reexamination counsel had scheduled an interview in the '128 reexamination to be held at the PTO on July 12th.

22. Had either Ms. Huttner or Mr. Jacobson specifically asked me about the status of the reexaminations after July 9th -- when I became aware that an interview was scheduled -- in the course of the settlement discussions, I would have informed them about the interview set for July 12th as that was the extent of my knowledge. The existence of a scheduled interview on July 12th had no impact on the agreed-to terms and provisions of the Settlement Agreement and it did not alter the status of the reexamination.

23. I first became aware of the interview summary issued by the PTO on July 25th when Ms. Huttner told me during a telephone call that it was posted on the PTO's Patent Application Information Retrieval website ("PAIR"). I obtained the interview summary from PAIR that day.

My Desire to Conclude the Settlement As Soon As Possible Was to Meet the Court's Deadlines in the Scheduling Order

24. Honeywell has accused Rohm and Haas of "surprisingly" acting quickly to complete the settlement agreement in an effort to get Honeywell to agree before it learned about the interview and the examiner's interview summary. This is wrong.

25. Rohm and Haas, mindful of the Court's Scheduling Order, wanted to complete the settlement agreement before the July 16, 2007 deadline set by the Court for the parties to submit a joint claim chart. The Scheduling Order in this case required the parties to submit a joint claim chart on June 1, 2007. (D.I. 23). To complete the settlement, the parties filed a joint stipulation to extend the deadline to file the joint claim chart from June 1st to July 2nd. (D.I. 55 Ex. A).

26. Needing more time to complete the settlement process, the parties filed two more joint stipulations extending the joint claim chart deadline ultimately to July 16th. (D.I. 72 and 73).

27. After the last request to extend the joint claim chart deadline (D.I. 73), the Court issued an oral order on July 10, 2007 stating that "the court WILL NOT entertain any additional stipulations to extend time regarding submission of the joint claim chart." Attached hereto as Exhibit O is a true and correct copy of the Court's July 10, 2007 oral order.

28. After receiving the Court's July 10th oral order, I immediately notified Ms. Huttner and Mr. Jacobson (as well as Ms. Votava of Honeywell and Mr. Frickey of Rohm and

Haas) by email that the Court would not grant any more extensions of time, and that we needed to complete the settlement as soon as possible. Attached hereto as Exhibit P is a true and accurate copy of my July 10th email.

29. On July 11th, Rohm and Haas was awaiting Honeywell's comments on the latest version of the settlement agreement and I sent an email to Honeywell's counsel that morning asking for their comments. Honeywell responded that they planned to respond later that day. At the end of the day, Rohm and Haas had not received any comments from Honeywell, so I sent another email to Honeywell's counsel asking about the status. In response, Mr. Jacobson stated that Honeywell expected to respond the next day. Mr. Jacobson added that he shared my desire to get the settlement done promptly. Attached hereto as Exhibit Q is a true and correct copy of my email exchange with Honeywell's counsel on July 11th.

30. Later on the evening July 11th, I emailed Ms. Huttner regarding the status of Honeywell's comments on the settlement agreement. Ms. Huttner replied that she would send me Honeywell's comments/changes the next day. Attached hereto as Exhibit R is a true and correct copy of my email exchange with Ms. Huttner on the evening of July 11th.

31. On July 12th Mr. Jacobson sent me Honeywell's changes to the agreement. Mr. Jacobson stated that Honeywell looked "forward to wrapping this up as soon as possible." Attached hereto as Exhibit S is Mr. Jacobson's July 12th email and the enclosed settlement agreement.

32. On July 13th, Mr. Jacobson sent an email stating that he would be out of the office from July 19, 2007 through July 30, 2007. Attached hereto as Exhibit T is a true and correct copy of Mr. Jacobson's July 13th email.

33. On July 13th, I received instructions from Rohm and Haas [REDACTED]

[REDACTED]

[REDACTED] As the window for completing the agreement before the July 16th deadline to submit a joint claim chart was closing, I called Ms. Huttner on the telephone to discuss the proposed change [REDACTED]. Ms. Huttner requested that I forward the proposed change to her by email. During our telephone call, both myself and Ms. Huttner expressed mutual concern that Honeywell may not approve Rohm and Haas's requested change. Attached hereto as Exhibit U is a true and correct copy of my July 13th email to Ms. Huttner with the proposed change to the covenant not to sue.

34. On the morning of July 16th, I participated in a teleconference with Rohm and Haas personnel to discuss Rohm and Haas's July 13th proposed change [REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]. Shortly thereafter, I sent Honeywell a version of the settlement agreement [REDACTED]

[REDACTED]

[REDACTED]. Attached hereto as Exhibit V is a true and correct copy of my email to Honeywell and the enclosed settlement agreement.

35. I also spoke with Ms. Huttner later on the morning of July 16th and advised her that Rohm and Haas no longer desired to substantially change [REDACTED]. In the evening of July 16th, I sent Honeywell's counsel an email stating:

I would like to call the Court tonite [sic] to leave a message that an agreement has been reached and that the parties will be working to execute the agreement tomorrow. Because were [sic, we are] to otherwise be filing a joint claim chart, as a courtesy to the Court, we should notify the Court what is going on regarding the settlement.

Ms. Huttner responded later that night that Honeywell would like to discuss the current draft the next day and that there were a few issues that she expected to be worked out. Attached hereto as Exhibit W is a true and correct copy of my email exchange with Honeywell on July 16th.

36. On July 17th, myself, Mr. Frickey, and Ms. Huttner participated in a teleconference where both Rohm and Haas and Honeywell concluded the final and complete Settlement Agreement. Ms. Huttner did say that she would have to get Mr. Jacobson to agree to the terms for Honeywell. Ms. Huttner did not ask about the status of the reexaminations during this teleconference. If she had asked, I would have advised her that an interview had been scheduled on July 12th, but that I was unaware of the substance of what happened at the interview.

37. Following the July 17th teleconference, Honeywell never suggested that it did not agree with all of the terms of the settlement agreement as written. To my knowledge, even today, Honeywell has not identified a single term in the agreement reached on July 17th that is inconsistent with the final agreement reached between the parties.

Honeywell Suggested The Parties Jointly Contact the Court to Announce the Settlement

38. On July 18th, Ms. Huttner emailed me suggesting that myself and her local co-counsel (Jim Taylor) contact the Court to announce that the parties have reached a settlement. I responded to Ms. Huttner that I would work it out with Mr. Taylor to contact the Court. I then telephoned Mr. Taylor and left him a voicemail. Mr. Taylor responded to my voicemail by email agreeing that the parties should jointly contact the Court and asked me if I knew when the signed agreement would be sent by Honeywell to Rohm and Haas. I replied back that I did not know when Honeywell would be sending Rohm and Haas the signed agreement. I asked Mr. Taylor when he would be available later that day to telephone the Court. In response, Mr. Taylor

suggested to call the Court immediately. Attached hereto as Exhibit X is a true and correct copy of the email exchange with Honeywell on July 18th.

39. During the parties' joint telephone call to the Court on July 18th, both myself and Mr. Taylor participated. Mr. Taylor initiated the call to the Court. We did not reach Chief Judge Sleet, but spoke to a member of his staff in his chambers. I identified the case and introduced the parties' representatives. I then said that both parties were calling to announce that a settlement agreement had been reached. I further said that the parties were exchanging signed agreements now and would be filing a joint stipulation of dismissal once that process was completed. I also said that the parties would be filing a joint letter to confirm what was said during the joint telephone call. I also apologized for the parties failure to file a joint claim chart on July 16th. Chief Judge Sleet's staff member responded that the Court had already marked in the file that the case was settled when the Parties previously informed the Court about the settlement.

40. When I asked Mr. Jacobson when he expected to get the Settlement Agreement signed on July 18th, he never stated that there was no final and binding agreement or that Honeywell disagreed with the terms of the Settlement Agreement. Nor did Mr. Jacobson express concern that both Rohm and Haas and Honeywell were going to jointly notify the Court that the parties had reached a final settlement. Mr. Jacobson also did not indicate that Honeywell's formal signature was a prerequisite to Honeywell's agreement to the terms of the July 17th Settlement Agreement.

41. During the settlement negotiations, it was my understanding that Mr. Jacobson had the full authority to speak for Honeywell and that Mr. Jacobson's agreement to the terms of the Settlement Agreement was Honeywell's agreement. It was my understanding that Ms. Huttner would have to confirm with Mr. Jacobson if Honeywell would agree to the terms of the

settlement agreement. I was not aware that Mr. Jacobson was allegedly unauthorized to bind Honeywell to any settlement agreement, nor did anyone so indicate.

42. At no point was I trying to conclude the settlement before the interview summary would be posted by the PTO on PAIR or otherwise known to Honeywell. I was unaware that the PTO had issued an interview summary. Further, I had no idea when an interview summary would be posted by the PTO on PAIR or otherwise transmitted to Honeywell. Rather, I was acting quickly out of respect for the Court's deadlines in its Scheduling Order, and in particular, the Court's oral order of July 10th which stated that the joint claim chart deadline would not be extended beyond July 16th. (*See* Paragraph 27 above). I was concerned that the settlement process would not be completed before certain filings were due with the Court, particularly since Mr. Jacobson announced on July 13th that he would be unavailable from July 19th through July 30th. Further, I was concerned that if a settlement was not reached, Rohm and Haas would be prejudiced by not meeting the Court's deadlines in the Scheduling Order.

43. Regretfully, we settled the case after the July 16th deadline to file the joint claim chart. When jointly contacting the Court on July 18th to advise the Court of the settlement, I apologized for not filing the joint claim chart on July 16th.

Honeywell Has Believed From the Beginning of this Dispute that Both of Rohm and Haas's '128 and '864 Patents Were Invalid

44. Honeywell answered Rohm and Haas's complaint on August 28, 2007 that both the '128 and '864 patents were invalid and void. (D.I. 10, ¶ 22). Honeywell also brought counterclaims alleging that the '128 and '864 patents were invalid and void and seeking a judicial declaration that the patents were invalid. (D.I. 10, ¶¶ 37 and 38). Attached hereto as

Exhibit Y is a true and correct copy of the relevant pages of Honeywell's Answer and Counterclaims (D.I. 10).

45. In response to Rohm and Haas's interrogatory number 6, Honeywell stated that, as of August 5, 2004, [REDACTED]. Attached hereto as Exhibit Z is a true and correct copy of Honeywell's response to Rohm and Haas's interrogatory no. 6.

46. Similarly, in December 2006, in support of its motion to stay the litigation pending the outcome of the reexaminations, Honeywell stated that it believed that all claims of the patents in suit will be invalidated after the reexamination is completed. Attached hereto as Exhibit AA is a true and correct copy of pages 8-9 of Honeywell's Memorandum of Law in Support of Its Motion to Stay Litigation Pending Reexamination of the Patents (D.I. 33).

47. In Honeywell's reply brief in connection with its motion to stay, Honeywell again pointed to its continued belief that both the '128 and '864 patents would be found invalid following the reexamination. Attached hereto as Exhibit BB is a true and correct copy of pages 1 to 3 of Honeywell's Reply Brief in Support of Its Motion to Stay Litigation Pending Reexamination of the Patents (D.I. 41).

48. Mr. Jacobson confirmed Honeywell's belief that the patents in suit were invalid in his declaration. (*See* Jacobson Decl. at ¶ 6).

In Spite of Honeywell's Belief that the '128 and '864 Patents Were Invalid, Honeywell Promised to Pay Rohm and Haas the Lump Sum Payment Regardless of the Outcome In the Reexamination

49. Despite Honeywell's consistently-stated belief that neither of the patents in suit would emerge from reexamination, [REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]. A true and correct copy of the June 26th draft settlement agreement is Exhibit K of my declaration. The final July 17th Settlement Agreement contained the same section [REDACTED]

[REDACTED]

[REDACTED]

Comparing the Interview Summary and the July 19th Supplemental Amendment with the June 11th Amendment Reveals that No Material Changes to the Claims Were Made No New Argument Was Raised and No PTO Action Occurred.

50. As stated above, upon request, I sent Honeywell a copy of the June 11, 2007 amendment in the '128 reexamination on June 20th. (*See* Paragraph 17 above, *see also* First Mulveny Decl. ¶ 5, Ex. D). This amendment made changes to original independent claims 1 and 5 as well as adding new claims 16 through 31. Attached hereto as Exhibit M is a true and correct copy of the June 11, 2007 amendment. The June 11th amendment also contains arguments distinguishing the claims over the cited references.

51. On July 19th, Mr. Corless (Rohm and Haas's reexamination counsel) filed a supplemental amendment in the '128 reexamination with the PTO. This amendment contained the same change to original independent claims 1 and 5 as well as adding the same new claims 16 through 31 that were part of the June 11, 2007 amendment. The only difference in the claims between the two amendments is the correction of typographical errors in new claims 21, 22, 23,

and a clarification to new claim 28. Attached hereto as Exhibit CC is a true and correct copy of the July 19, 2007 amendment.

52. Honeywell was aware of the amendments to the original claims of the '128 patent when I sent the June 11th amendment to Ms. Huttner. There has been no change to the original claims since the June 11th amendment.

53. Honeywell admits that settlement talks broke down after June 19th. (*See* Jacobson Decl. ¶ 21). Honeywell further admits that it returned to the settlement table after I had sent Ms. Huttner a copy of the June 11th amendment filed in the '128 patent reexamination. (*See* Jacobson Decl. ¶¶ 22 and 23). Thus, Honeywell has constantly been aware of all of the changes to the claims in the '128 patent reexamination (with the exception of the corrected typographical errors in new claims 21, 22, and 23, and a clarification to new claim 28).

54. I have compared the interview summary with the June 11th amendment. According to the interview summary, Rohm and Haas's reexamination counsel made the same arguments that were presented in the June 11th amendment. I have reviewed the interview summary and it is not material to the patentability of the '128 patent claims as no new rejections were made, nor was any pending rejection made final. The interview summary was a non event.

Ms. Huttner Failed to Properly Change Her Address in the Reexaminations

55. While Ms. Huttner apparently changed her address in the PTO Attorney's Roster (*see* Huttner Decl. at ¶ 4), there is no record establishing that she filed a change of correspondence address in either the '128 or '864 reexaminations as required by the PTO. Attached hereto as Exhibit DD is a true and correct copy of the PTO Manual of Patent Examining Procedure (MPEP) § 601.03, page 600-24. The MPEP states (emphasis added):

Unless the correspondence address is designated as the address associated with a Customer Number, a separate notification *must be filed in each application for which a person is intended to receive communications from the Office.*

* * *

The obligation (see 37 CFR 11.11) of a registered attorney or agent to notify the Attorney's Roster by letter of any change of his or her address for entry on the register *is separate from the obligation to file a notice of change of address filed in individual applications.* See MPEP § 402.

56. I have reviewed the transaction histories and image file wrappers on PAIR for both the '128 and '864 reexaminations and have found no record that Ms. Huttner filed a separate change of address in each reexamination as required by the PTO for her to receive communications from the PTO.

I Did Not Realize Ms. Huttner Had Not Changed Her Correspondence Address

57. I was unaware that Rohm and Haas's '128 reexamination papers were being sent to Ms. Huttner's former address at Skadden until Honeywell sent its letter to the Court on July 26th. (D.I. 75). In its July 26th letter, Honeywell disclosed that the interview summary was sent to Ms. Huttner's incorrect address. (D.I. 75, fn.1). Seeing this, I obtained the complete interview summary from PAIR and observed that the PTO was using Ms. Huttner's former Skadden address. I then reviewed Rohm and Haas's June 11th amendment on PAIR and also discovered that it also was sent to Ms. Huttner's former Skadden address. The July 19th supplemental

amendment was filed before I became aware that Rohm and Haas's '128 reexamination papers were being sent to Ms. Huttner's former Skadden address, thus I could not provide instructions to Mr. Corless to send the July 19th supplemental amendment to Ms. Huttner's new address.

I declare under penalty of perjury that the foregoing is true and correct to the best of my knowledge and belief.

Executed at Wilmington, Delaware, on September 4, 2007.

/s/ Daniel C. Mulveny

Daniel C. Mulveny, Esq. (#3984)
CONNOLLY BOVE LODGE & HUTZ LLP
The Nemours Building
1007 North Orange Street
P.O. Box 2207
Wilmington, DE 19899
(302) 658-9141

560716_1.DOC

TABLE OF CONTENTS

| <u>Document</u> | <u>Exhibit</u> |
|--|----------------|
| Ex Parte Reexamination Response to Office Action for U.S. Patent App. SN 90/008,359 [corresponding to the '128 patent] (June 11, 2007)..... | M |
| Email from Scott Jacobson to Daniel C. Mulveny et al. (June 29, 2007 10:22 AM)..... | N |
| Order by the Court (July 10, 2007) | O |
| Email from Daniel C. Mulveny to Scott Jacobson et al. (July 10, 2007 11:10AM)..... | P |
| Email exchange between Daniel C. Mulveny and Honeywell (July 11, 2007 5:51 PM) | Q |
| Email exchange between Daniel C. Mulveny and Honeywell (July 11, 2007 8:57 PM) | R |
| Email from Scott Jacobson to Daniel C. Mulveny et al. (July 12, 2007 4:51 PM) and enclosed draft settlement agreement | S |
| Email from Scott Jacobson to Daniel C. Mulveny et al. (July 13, 2007 5:46 PM) | T |
| Email from Daniel C. Mulveny to Constance Huttner (July 13, 2007 2:17 PM) | U |
| Email from Daniel C. Mulveny to Scott Jacobson et al. (July 16, 2007 10:51 AM) and enclosed draft settlement agreement | V |
| Email from Daniel C. Mulveny to Scott Jacobson et al. (July 16, 2007 6:32 PM) | W |
| Email exchange between Daniel C. Mulveny and Honeywell (July 18, 2007 10:13 AM)..... | X |
| Relevant pages of Honeywell's Answer and Counterclaims (D.I. 10) | Y |
| Honeywell's Response to Rohm and Haas's Interrogatory No. 6..... | Z |

| | |
|---|----|
| Relevant pages of Honeywell's Memorandum of Law in Support of Its Motion to Stay Litigation Pending Reexamination of the Patents (D.I. 33) | AA |
| Relevant pages of Honeywell's Reply Brief in Support of Its Motion To Stay Litigation Pending Reexamination of the Patents (D.I. 41)..... | BB |
| Ex Parte Reexamination Supplemental Amendment for U.S. Patent App. SN 90/008,359 [corresponding to the '128 patent] (July 19, 2007)) | CC |
| PTO Manual of Patent Examining Procedure (MPEP) § 601.03, page 600-23 to 600-24 | DD |

CERTIFICATE OF SERVICE

I hereby certify that on September 4, 2007, I electronically filed Second Declaration of Daniel C. Mulveny, Esq. (Filed in Support of Plaintiff Rohm and Haas Electronic Materials LLC's Reply Memorandum on Its Motion to Enforce the Settlement Agreement) with the Clerk of the Court using CM/ECF which will send notification of such filing to the following:

Email & Hand Delivery

James D. Taylor, Jr.
Buchanan Ingersoll & Rooney PC
The Brandywine Building
1000 West Street, Suite 1410
Wilmington, DE 19801

Email & FedEx

Constance S. Huttner
Buchanan Ingersoll & Rooney PC
One Chase Manhattan Plaza, 35th Floor
New York, NY 10005-1417

/s/ Daniel C. Mulveny
Rudolf E. Hutz (#484)
Daniel C. Mulveny (#3984)
1007 N. Orange Street
P. O. Box 2207
Wilmington, DE 19899-2207
(302) 658-9141
Attorneys for Plaintiff

EXHIBIT M

Express Mail Label No. EV 971745846US
Docket No.: 40678-5C
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application:
Thackeray et al.

Application No.: 90/008,359

Confirmation No.: 8757

Filed: December 4, 2006

Art Unit: 3991

For: ANTIHALATION COMPOSITIONS

Examiner: S. Stein

AMENDMENT

MS *Ex Parte* Reexam
ATTN: Central Reexamination Unit
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

INTRODUCTORY COMMENTS

This document is submitted in response to the Office Action mailed from the United States Patent and Trademark Office on April 9, 2007 in the above-identified application.

Amendments to the Claims are reflected in the listing of claims which begins on page 2 of this paper.

Remarks begin on page 6 of this paper.

Application No. 90/008,359

2

Docket No.: 40678-5C

Amendments to the Claims

Claim 1. (amended) A coated substrate comprising:
a substrate having thereon:
a coating layer of an antireflective composition, the antireflective composition comprising a crosslinker and an anthracene material; and
a coating layer of a positive-acting photoresist composition over the antireflective composition coating layer.

Claim 2. (original) The substrate of claim 1 wherein the antireflective composition coating layer is crosslinked.

Claim 3. (original) The substrate of claim 1 wherein the antireflective composition comprises a thermal acid generator.

Claim 4. (original) The substrate of claim 1 wherein the substrate is a microelectronic wafer.

Claim 5. (amended) A method for forming a relief image on a substrate comprising:
applying on the substrate a layer of an antihalation composition comprising an anthracene material;
applying over the antihalation composition coating layer a positive-acting photoresist composition.

Claim 6. (original) The method of claim 5 wherein the antihalation composition coating layer is crosslinked prior to applying the photoresist composition.

Application No. 90/008,359

3

Docket No.: 40678-5C

Claim 7. (original) The method of claim 6 wherein the photoresist composition is imaged with activating radiation and the imaged photoresist composition is treated with a developer to provide a photoresist relief image.

Claim 8. (original) The method of claim 7 wherein areas bared of photoresist upon treatment with the developer are etched.

Claim 9. (original) The method of claim 7 wherein areas bared of photoresist upon treatment with the developer are exposed to a plasma gas.

Claim 10. (original) The method of claim 9 wherein the plasma gas penetrates the antihalation composition coating layer.

Claim 11. (original) The method of claim 5 wherein the antihalation composition comprises a thermal acid generator.

Claim 12. (original) The method of claim 5 wherein the substrate is a microelectronic wafer.

Claim 13. (original) The method of claim 5 wherein the photoresist composition is imaged with activating radiation and the imaged photoresist composition is treated with a developer to provide a photoresist relief image.

Claim 14. (original) The method of claim 13 wherein areas bared of photoresist upon treatment with the developer are etched.

Claim 15. (original) The method of claim 13 wherein areas bared of photoresist upon treatment with the developer are exposed to a plasma gas.

Claim 16. (original) The method of claim 15 wherein the plasma gas penetrates the antihalation composition coating layer.

Application No. 90/008,359

4

Docket No.: 40678-5C

Claim 17. The method of claim 6 wherein the photoresist composition is imaged with radiation having a wavelength of 100 nm to 300 nm.

Claim 18. The method of claim 6 wherein the photoresist composition is imaged with radiation having a wavelength of 248 nm.

Claim 19. The method of claim 7 wherein the photoresist layer is imaged with radiation having a wavelength of 100 to 300 nm.

Claim 20. The method of claim 7 wherein the photoresist layer is imaged with radiation having a wavelength of 248 nm.

Claim 21. The method of claim 5 wherein the positive composition is a chemically amplified positive-acting photoresist composition.

Claim 22. The method of claim 18 wherein the positive composition is a chemically amplified positive-acting photoresist composition.

Claim 23. The substrate of claim 1 wherein the positive composition is a chemically amplified positive-acting photoresist composition.

Claim 24. A method for forming a relief image on a substrate comprising:
applying on the substrate a layer of an antihalation composition comprising an anthracene material;
applying over the antihalation composition coating layer a photoresist composition; and
exposing the applied photoresist composition to patterned radiation having a wavelength of 248 nm.

Claim 25. The method of claim 25 wherein the antihalation composition is crosslinked prior to applying the photoresist composition over the antihalation composition layer.

Application No. 90/008,359

5

Docket No.: 40678-5C

Claim 26. The method of claim 24 wherein the photoresist composition is a chemically-amplified positive-acting photoresist composition.

Claim 27. The method of claim 25 wherein the photoresist composition is a chemically-amplified positive-acting photoresist composition.

Claim 28. A method for forming a relief image on a substrate comprising:
applying on the substrate a layer of an antihalation composition comprising an anthracene material;
crosslinking the antihalation composition layer; and
applying over the antihalation composition coating layer a photoresist composition.

Claim 29. The method of claim 28 further comprising imaging the photoresist composition with activating radiation and treating the imaged photoresist composition with a developer to provide a photoresist relief image.

Claim 30. The method of claim 29 wherein areas bared of photoresist upon treatment with the developer are etched.

Claim 31. The method of claim 29 wherein areas bared of photoresist upon treatment with the developer are exposed to a plasma gas.

Application No. 90/008,359

6

Docket No.: 40678-5C

REMARKS

Claims 1-31 are pending herein. Claims 1 and 5 have been amended, and claims 17-31 have been added. No new matter has been added by those amendments. For instance, support for the amendments of claims 1 and 5 appears e.g. at page 17, lines 11-15 of the application. Support for the new claims appears e.g. page 15, line 31 through page 16, line 1; page 19, lines 3-16 and page 21, lines 29-32; pages 26-27; and the original claims of the application as filed.

The new claims also do not enlarge the scope of the issued patent claims. Thus, new independent claims 24 and 28 recite at least the subject matter of patent claim 5.

Claims 7, 11, 12 and 15 were rejected under the doctrine of obviousness-type double patenting over certain claims of U.S. Patent 5,851,738.

To expedite matters, an appropriate Terminal Disclaimer is submitted herewith. Withdrawal of the rejection is requested.

Claims 1-5 and 11-14 were rejected under 35 U.S.C. 102 over Rhode (U.S. Patent 4,935,320). The rejection is traversed.

Claims 1 and 5 (the only rejected independent claims) each call for a "positive-acting photoresist."

The Rhode document does not describe use of positive photoresists. Rather, the Rhode document describes use of a negative composition only. See, for instance, column 32 of Rhode.

Accordingly, the rejection is properly withdrawn. See *In re Marshall*, 198 USPQ 344, 346 (CCPA 1978) ("[r]ejections under 35 U.S.C. §102 are proper only when the claimed subject matter is identically disclosed or described in the prior art.").

Application No. 90/008,359

7

Docket No.: 40678-5C

Claims 1, 2, 4-8, 12 and 13 were rejected under 35 U.S.C. 102 over Jain (U.S. Patent 4,863,827).

As the rejection is understood, Jain is cited for a report for certain anthraquinone compounds, and such anthraquinone compounds are considered to constitute an anthracene compound.

The rejection is traversed.

It is well-recognized that an anthraquinone compound is not an anthracene compound. See, for instance, Hawley's Condensed Chemical Dictionary, 11th edition, pages 85-86, copy enclosed. This is consistent with the present application, such as at page 12, line 24 through page 13, line 18 of the specification.

In view thereof, reconsideration and withdrawal of the rejection are requested. See *In re Marshall, supra*.

Claims 5 and 12 were rejected under 35 U.S.C. 102 over Koshimo (U.S. Patent 3,888,702). As the rejection is understood, Koshimo is cited for a report of an anthroquinone violet, which is indicated in the Office Action to constitute an anthracene compound. The rejection is traversed.

This rejection suffers from deficiencies noted above. Anthroquinone violet dye contains anthraquinone compounds, which are not anthracene compounds. See Jaskot et al., *Toxicological Sciences*, 22(1):103-112 (1994), copy enclosed.

In view thereof, reconsideration and withdrawal of the rejection are requested.

Claims 5 and 12-16 were rejected under 35 U.S.C. 103 over Arnold (U.S. Patent 4,910,122) in view of Koshimo (U.S. Patent 3,888,702). The rejection is traversed.

Application No. 90/008,359

8

Docket No.: 40678-5C

The Arnold document does not remedy the shortcomings of Koshimo as discussed above and incorporated by reference here.

Thus, among other things, neither Koshimo nor Arnold reports use of an anthracene material. In the Office Action 10, it is specifically admitted that "Arnold does not specifically disclose the use of an anthracene in the antireflective layer." As discussed above, the report of anthraquinone violet in Koshimo is not a disclosure of an anthracene material.

Accordingly, withdrawal of the rejection is requested.

Claims 9 and 10 were rejected under 35 U.S.C. 103 over Jain (U.S. Patent 4,863,827) and further in view of Arnold (U.S. Patent 4,910,122). The rejection is traversed.

Again, the addition of the Arnold document does not remedy the shortcomings of Jain as discussed above and incorporated by referenced here.

Thus, among other things, neither Jain nor Arnold reports use of an anthracene material. As noted, in the Office Action at page 10, it is specifically admitted that "Arnold does not specifically disclose the use of an anthracene in the antireflective layer." As discussed above, the report of anthraquinone compound in Jain is not a disclosure of an anthracene material.

Withdrawal of the rejection is requested.

It is believed the application is in condition for immediate allowance, which action is earnestly solicited.

Application No. 90/008,359

9

Docket No.: 40678-5C

Respectfully submitted,

By 

Peter F. Corless

Registration No.: 33,860

EDWARDS ANGELL PALMER & DODGE
LLP

P.O. Box 55874

Boston, Massachusetts 02205

(617) 439-4444

Attorneys/Agents For Applicant

PTO/SB/25 (09-08)

Approved for use through 03/31/2007. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

**TERMINAL DISCLAIMER TO OBTAIN A DOUBLE PATENTING
REJECTION OVER A "PRIOR" PATENT**

Docket Number (Optional)

40678REX(70329)

In re Application of: James W. Thackeray et al.

Application No.: 90/008,359

Filed: December 4, 2006

For: ANTIHALATION COMPOSITIONS

The owner*, Shibley Company, L.L.C., of 100 percent interest in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of prior patent No. 5,851,738 as the term of said prior patent is defined in 35 U.S.C. 154 and 173, and as the term of said prior patent is presently shortened by any terminal disclaimer. The owner hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and the prior patent are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.

In making the above disclaimer, the owner does not disclaim the terminal part of the term of any patent granted on the instant application that would extend to the expiration date of the full statutory term as defined in 35 U.S.C. 154 and 173 of the prior patent, "as the term of said prior patent is presently shortened by any terminal disclaimer," in the event that said prior patent later:

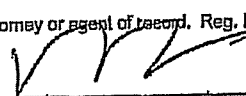
- expires for failure to pay a maintenance fee;
- is held unenforceable;
- is found invalid by a court of competent jurisdiction;
- is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321;
- has all claims canceled by a reexamination certificate;
- is reissued; or
- is in any manner terminated prior to the expiration of its full statutory term as presently shortened by any terminal disclaimer.

Check either box 1 or 2 below, if appropriate.

1. ☐ For submissions on behalf of a business/organization (e.g., corporation, partnership, university, government agency, etc.), the undersigned is empowered to act on behalf of the business/organization.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

2. ☒ The undersigned is an attorney or agent of record. Reg. No. 33,860



Signature

June 11, 2007

Date

Peter F. Corless

Typed or printed name

(617) 517-5557

Telephone Number

- ☒ Terminal disclaimer fee under 37 CFR 1.20(d) is included.

*Statement under 37 CFR 3.73(b) is required if terminal disclaimer is signed by the assignee (owner).
Form PTO/SB/96 may be used for making this certification. See MPEP § 324.

*Hawley's
Condensed Chemical
Dictionary*


ELEVENTH EDITION

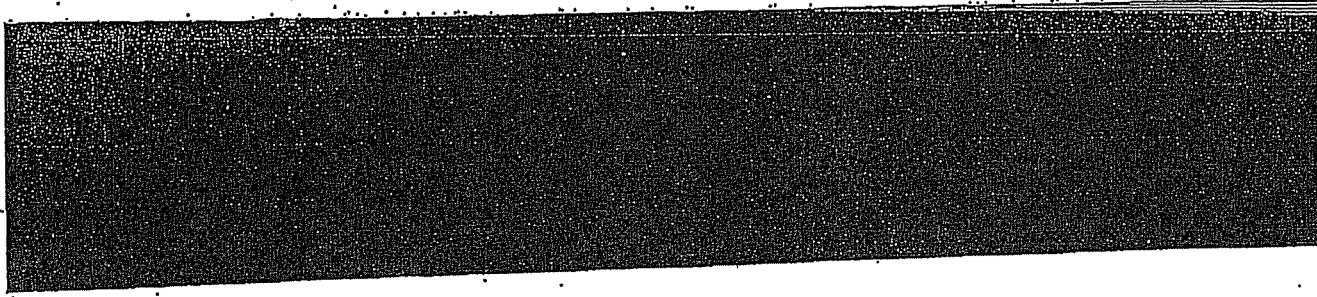
Revised by

N. Irving Sax

and

Richard J. Lewis, Sr.

 VAN NOSTRAND REINHOLD
New York



development. Such biologicals between sulfa drugs and antibiotics, and also between histamine compounds collectively.

One of the most important cultural standpoint is imidazole with similar compounds, the metabolism of insects, king fabrics; it is also being used to protect plants from natural antagonists have implications in the complex field

anticoagulant, antigen-antibody

TM for a high-foaming, water-soluble surfactant with soap-like fatty amido compound 40%

1 detergent for woolen and silk under neutral, acid, and alkali recommended for use in bubble soaps for dedusting purposes

a series of surfactants, additive, preventing accumulation pipe lines, reels, etc. Prevents in manufacture of deposits from forming on rollers, in the steel industry, etc in the final wash of the cold reduction.

colorless crystalline powder 120°C. Sparingly soluble in water, practically insoluble in benzene.

stamine). Available as hydrophosphate.

ring an isomer (usually a dimer) which has a single, amide at the third carbon from the chain in distinction to anthracene the attachment is to the end. For example isododecane, while anteisododecane, $H_2, COOH$.

at used in veterinary medicine

name for an antibiotic substance Streptomyces longissimus.

antheridiol, $C_{25}H_{42}O_6$.

Properties: Colorless, fine crystals; mp 250°C; slightly soluble in water, soluble in warm methanol.

Use: A plant hormone having a specific sex function, it is secreted by certain water molds. It has been used to modify plant fertility. Said to be the first plant sex hormone to be discovered (1942).

anthocyanin. A flavonoid plant pigment which accounts for most of the red, pink, and blue colors in plants, fruits, and flowers. Water-soluble. See also flavonoid.

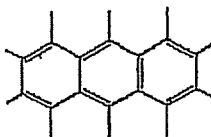
"Anthomine."TM TM for a dyeing assistant primarily for use in wool-dyeing.

anthophyllite. $(Mg, Fe)_7Si_8O_{22}(OH)_2$. A natural magnesium-iron silicate. See asbestos.

anthracene. CAS: 120-12-7.

$C_{14}H_{10}$ (CH)₂ C_6H_4 .

Properties: Yellow crystals with blue fluorescence. Soluble in alcohol and ether, insoluble in water, d. 1.25 (27/4°C), mp 217°C, bp 340°C, sp 250F (121°C) (CC). Combustible. It has semiconducting properties.



Derivation: (a) By salting out from crude anthracene oil and draining. The crude salts are purified by pressing and finally by the use of various solvents. Phenanthrene and carbazole are removed; (b) by distilling crude anthracene oil with alkali carbonate in iron retorts, the distillate containing only anthracene and phenanthrene. The latter is removed by carbon disulfide.

Method of purification: By sublimation with superheated steam or by crystallization from benzene followed by sublimation; for very pure crystals, zone melting of solid anthracene. Impurities: Phenanthrene, carbazole, and chrysene.

Grade: Commercial (90-95%), pure crystals.

Hazard: A carcinogen.

Use: Dyes, alizarin, phenanthrene, carbazole, anthraquinone, calico printing, a component of smoke screens, scintillation counting crystals, organic semi-conductor research.

anthracene oil. A coal-tar fraction boiling in the range 270-360°C, a source of anthracene and similar

ilar aromatics. Also used as a wood preservative and pesticide, except on food crops. Hazard: A carcinogen.

1,8,9-anthracenetriol. See anthralin.

anthracite. See coal.

anthragallic acid. See anthragalloi.

anthragalloi. (1,2,3-trihydroxyanthraquinone; anthragallic acid). $C_{14}H_8(OH)_3(CO)_2$.

Tricyolic.

Properties: Brown powder. Soluble in alcohol, ether, glacial acetic acid, slightly soluble in water and chloroform. Sublimes at 290°C.

Derivation: Product of the reaction of benzole, gallic, and sulfuric acids.

Use: Dyeing.

"Anthragen."TM TM for a series of lake colors. Used for printing inks, wallpaper, coated paper, paint, rubber, and organic plastics.

"Anthralin."TM TM for a series of acid dyes. Used on wool.

anthralin. (1,8,9-anthracenetriol; 1,8-dihydroxyanthranol). $C_{14}H_{10}O_3$.

Properties: Odorless, tasteless, yellow powder. Mp 176-181°C. Filtrate from water suspension is neutral to litmus. Soluble in chloroform, acetone, benzene, and in solutions of alkali hydroxide; slightly soluble in alcohol, ether, and glacial acetic acid; insoluble in water. Combustible.

Derivation: By catalytic reduction of 1,8-dihydroxyanthraquinone with hydrogen at high pressure.

Grade: NF, (95%).

Hazard: Very irritating. Do not use on scalp or near eyes.

Use: Medicine (treatment of psoriasis).

anthranilic acid. (o-aminobenzoic acid). CAS: 118-92-3. $C_6H_4(NH_2)(CO_2H)$.

Properties: Yellowish crystals; sweetish taste; soluble in hot water, alcohol, and ether. Mp 144-146°C, sublimes. Combustible.

Derivation: Phthalimide plus an alkaline hypobromite solution.

Grade: Technical (95-98%), 99% or better.

Use: Dyes, drugs, perfumes, and pharmaceuticals.

anthranol. (9-hydroxyanthracene).

$C_{14}H_{10}OH$.

Properties: Crystals, mp 120°C, soluble in organic solvents with a blue fluorescence. Changes in solution to anthrone. Combustible.

Use: Dyes.

ANTHRANONE

86

anthranone. See anthrone.

"Anthrapole,"TM for a group of dye carriers or assistants for use in dyeing polyester fibers and blends. Active ingredients are aromatic esters, chlorinated hydrocarbons, and phenol derivatives. Emulsifying agents are incorporated.

anthrapurpurin. (1,2,7-trihydroxyanthraquinone; isopurpurin; purpurin red).

$C_{14}H_8O_5$. Tricyclic.

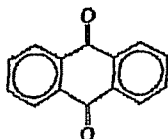
Properties: Orange-yellow needles. Soluble in alcohol and alkalies, slightly soluble in ether and hot water. Mp 369°C, bp 462°C.

Derivation: By fusion of anthraquinonedisulfonic acid with caustic soda and potassium chlorate; the melt is run into hot water and the anthrapurpurin precipitated by hydrochloric acid.

Use: Dyeing, organic synthesis.

anthraquinone. CAS: 84-65-1.

$C_{14}H_8O_2$.



Properties: Yellow needles. Soluble in alcohol, ether, and acetone; insoluble in water; d 1.419-1.438, mp 286°C, bp 379-381°C, flash p 365°F (185°C) (CC). Combustible.

Derivation: (a) By heating phthalic anhydride and benzene in the presence of aluminum chloride and dehydrating the product; (b) by condensation of 1,4-naphthoquinone with butadiene.

Method of purification: Sublimation.

Grade: Sublimed, 30% paste (solid on 100% basis), electrical 99.5%.

Use: Intermediate for dyes and organics, organic inhibitor, bird repellent for seeds. See also anthraquinone dye.

anthraquinone-1,5- and 1,8-disulfonic acids.

(rho acid, chi acid) respectively.

$C_{14}H_8O_6S_2$.

Properties: (In their pure state) Slightly yellow to white crystals. The technical grade is grayish-white. Soluble in water and strong sulfuric acid. The 1,8-isomer is the more soluble. The 1,5-disulfonic acid melts with decomposition at 310-311°C. The 1,8-isomer melts and decomposes at 293-294°C.

Derivation: Anthraquinone is sulfonated with fuming sulfuric acid in the presence of mercury or mercuric oxide to a mixture of the 1,5- and

1,8-disulfonic acids which are separated by fractional crystallization.

Method of purification: Fractional crystallization from strong sulfuric acid or in form of their alkali salts from either acid or alkaline solutions.

Grade: Technical.

Use: Dyes.

anthraquinone dye. A dye whose molecular structure is based on anthraquinone.

$C_{14}H_8(CO)_2C_6H_4$.

The chromophore groups are $=C=O$ and $=C=C=$. The benzene ring structure is important in the development of color. CI numbers from 58000 to 72999. These are acid or mordant dyes when OH or HSO_3 groups respectively are present. The anthraquinone dyes that can be reduced to an alkaline solution (leuco (vat) derivative that has affinity for fibers and which can be reoxidized to the dye are known as anthraquinone vat dyes. They are largely used on cotton, rayon, and silk, and have excellent properties of color and fastness.

anthracene. See 1,5-dihydroxyanthraquinone.

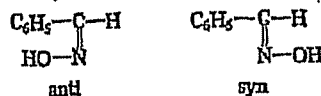
anthrone. (anthranone; 9,10-dihydro-9-oxoanthracene). $C_{14}H_{10}O$. The keto is the more stable form of anthranol.

Properties: Colorless needles, mp 156°C, insoluble in water, soluble in alcohol, benzene, and hot sodium hydroxide solutions.

Derivation: Reduction of anthraquinone with tin and hydrochloric acid.

Use: Rapid determination of sugar in body fluids, and of animal starch in liver tissue; general reagent for carbohydrates; organic synthesis.

anti-. (1) A prefix used in designating geometrical isomers in which there is a double bond between the carbon and nitrogen atoms. This prevents free rotation so that two different spatial arrangements of substituent atoms or groups are possible. When a given pair of these are on opposite sides of the double bond, the arrangement is called anti-; when they are on the same side, it is called syn-, as indicated below:



These prefixes are disregarded in alphabetizing chemical names. (2) A prefix meaning "against," or "opposed to," as an antibody, antimalarial, etc.

anxiolytic agent. See psychotropic drug.

FUNDAMENTAL AND APPLIED TOXICOLOGY 22, 103-112 (1994)

Toxicity of an Anthraquinone Violet Dye Mixture Following Inhalation Exposure, Intratracheal Instillation, or Gavage¹

RICHARD H. JASKOT* AND DANIEL L. COSTA†

*ManTech Environmental Technologies, Inc., Research Triangle Park, North Carolina 27709; and †U.S. Environmental Protection Agency, Health Effects Research Laboratory, Research Triangle Park, North Carolina 27711

Received March 10, 1992; accepted September 16, 1993

Toxicity of an Anthraquinone Violet Dye Mixture Following Inhalation Exposure, Intratracheal Instillation, or Gavage. JASKOT, R. H., AND COSTA, D. L. (1994). *Fundam. Appl. Toxicol.* 22, 103-112.

Anthraquinone dyes are utilized by the military in colored-smoke grenades. During production, workers in munitions plants may be exposed to fugitive emissions of these dyes or mixtures thereof. The effects of a prototype violet dye mixture (VDM) consisting of Disperse Red 11 (DR11), [1,4-diamino-2-methoxy-anthraquinone] and Disperse Blue 3 (DB3) [1-methylamino-4-hydroxyethylamino-anthraquinone] on F344 male and female rats have been investigated. Acute 1-day inhalation exposures (6 hr) to VDM were conducted at 1000, 300, 100, 70, 40, and 10 mg/m³, with an additional exposure to 40 mg/m³ 6 hr/day for 5 days; 4.22 ± 2.1 µm (MMAD ± σg). Lung burdens of dye, general histopathology, and/or liver function were evaluated at 0, 3, and 7 days postexposure. Unexpected lethality due to severe liver damage was observed with acute exposures of >300 mg/m³ and in the 5-day 40 mg/m³ exposures. Centrilobular degeneration and necrosis of liver cells was concentration-dependent with inhalation of VDM ≥40 mg/m³. In addition, nasal olfactory epithelium exhibited degeneration and necrosis with acute exposures ≥10 mg/m³. Lung instillations at 250, 500, and 1000 µg of the VDM revealed no lung or liver toxicity. Because per os exposure due to preening was suspected as a major exposure route, a gavage study with the VDM and its two component dyes DR11 and DB3 (800 mg/kg) was undertaken. One day following gavage with DR11 or DB3, serum enzymes indicative of liver toxicity (LDH, SGPT, SDH, and ICDH) were slightly elevated (1-6X control). However, rats gavaged with VDM had serum enzyme levels 10-100X control by Day 1 after gavage, indicating acute liver toxicity. Activities of liver enzymes involved in xenobiotic and glutathione metabolism were also acutely affected. All of the dyes caused various degrees of induction of glucose-6-phosphate dehydrogenase, glutathione

reductase, glutathione peroxidase, and nonprotein sulfhydryls. The enzymes involved in xenobiotic metabolism (glutathione S-transferase, NADPH cytochrome-c reductase, and P450) were also elevated by the two component dyes, in contrast to their significant depression with VDM treatment. The similarity between the liver and olfactory epithelium effects of these compounds and the lack of pulmonary tissue effects is not fully understood, but the interaction of the individual dyes as VDM emphasizes the need to assess chemicals such as the anthraquinones as their likely-to-be-encountered mixtures. © 1994 Society of Toxicology.

Anthraquinones are a diverse group of naturally occurring and synthetic chemical compounds used widely in industry as colorants in foods, drugs, cosmetics, hair dyes, and textiles and in medicine as purgative, antimicrobial, and antitumor preparations (Sendelbach, 1989). In general, little information is available concerning the health risks of most anthraquinones, and human toxicity data are lacking except for a few clinical reports on allergic contact dermatitis (NIOSH, 1981; Hatch, 1984).

The U.S. Army utilizes various anthraquinone dyes in colored-smoke grenades for marking, signaling, and identification of sites in the field. The dye is purchased from commercial suppliers and is formulated with a strong oxidant and other combustion enhancers into combustible mixtures within grenades in what are described as load, assembly, and pack (LAP) plants. The nature of these dye compounds makes full containment difficult in grenade manufacture and hence incidental worker exposure is virtually unavoidable; contact may be via inhalation or dermal exposure. Additional concern is raised by the potential exposure of ground troops during training and field operations.

Inhalation toxicity studies on two dyes similarly used in colored-smoke munitions, Solvent Yellow 33 (a quinoline dye) and Solvent Green 3 (an anthraquinone dye), have previously been reported (Sun *et al.*, 1987). Both the yellow dye and yellow/green dye mixture exhibited low toxicity, although the yellow/green mix appeared slightly more toxic than the yellow dye alone, suggesting some synergistic in-

¹ Disclaimer: This report has been reviewed by the Health Effects Research Laboratory, U.S. Environmental Protection Agency, and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use. Funding for this work was provided by the U.S. Army Medical Research and Development Command (USAMRDC), Project 87PF7808.

interaction between the two dyes. Another dye mixture, a prototype violet dye, with advantageous physical and chemical properties for use in smoke munitions, was proposed for widespread use and was evaluated in the present study. This binary mixture consisted of two pure anthraquinone dyes, 92.9% Disperse Red 11 (DR11) [1,4-diamino-2-methoxy-anthraquinone] and 7.1% Disperse Blue 3 (DB3) [1-methylamino-4-hydroxyethylamino-anthraquinone] (Fig. 1). The only available toxicity data on these components indicated that the acute oral LD50 values was in the range of 2–3 g/kg (Material Data Safety Sheet: DR11, 86-0608, DB3; 86-0056) and, in the case of violet dye mixture (VDM), 794–1000 mg/kg and 1413–1778 mg/kg for undefined male and female rats, respectively (J. Eaton, USAMRDC, personal communication). The comparability of these LD50 assays for the component and mixed dyes, and hence the comparability of the LD50 values themselves, is questionable. Inhalation data providing evidence of toxicity are not available.

The present study was designed to evaluate the biological impact of this prototype VDM on small laboratory animals in order to assess the potential health risks to humans incidentally exposed in the handling of the dye mixture in the workplace. This report presents the findings of several studies to characterize the toxicity of VDM in rats, including studies which involved exposures by inhalation (whole-body and nose-only), gavage, and intratracheal instillation of VDM and, in selected experiments, the component dyes (DR11 and DB3).

MATERIALS AND METHODS

Animals. Ninety-day-old, Fischer 344 SPF male and female rats (Charles River Laboratories, Raleigh, NC) were used throughout the study. The rats were received at 60 days of age and held for 30 days prior to exposure. The animals were housed by gender 3 per cage and maintained under conditions of constant temperature ($72 \pm 2^\circ\text{F}$), humidity ($50 \pm 10\%$), and lighting (12:12 light:dark cycle) until they were exposed. Purina Rat Chow 5001 and water were provided *ad libitum* with only water available during exposures. Each study group (concentration) consisted of 18 to 32 rats evenly divided by gender unless otherwise noted in the text or tables. Because the concentration-response studies did not reveal gender-based susceptibility, follow-up studies used only male rats in an attempt to minimize variability in the findings.

VDM aerosol exposure system. A detailed description of the aerosol exposure system, including aerosol generation and monitoring, chamber distribution, and particle characterization, is presented elsewhere (Higuchi

and Steinhagen, 1991; Higuchi and Davies, 1990). Briefly, the aerosol exposure system for generation of VDM consisted of modified dry material feeder (AccuRate, Model 106), jet grinding mill (Jet-o-Mizer, Model 0101), single-stage impactor, aerosol neutralizer, and sound-insulating foam. The powder feeder delivered the dye through a venturi on the jet mill grinding chamber, where two high-speed air jets ground the bulk powder material into the finer particles. The ground dye exited the jet mill and entered a single-stage impactor where the aerosol stream was neutralized to eliminate electrostatic charge on the dye particles, mixed with humidified dilution air, and passed through a tubular section of sound-dampening foam, to reduce both high- and low-frequency noise created by the jet mill, before entering the inhalation chambers.

Four 2-m³ stainless steel/glass exposure chambers (Hazleton-2000) were used to conduct three concentration and one clear air control exposures simultaneously. The inhalation chambers were monitored continuously for temperature ($72 \pm 2^\circ\text{F}$) and relative humidity ($60 \pm 20\%$). Additional monitoring included chamber air flow (at least 15 air changes/hr), contaminant flow rate (mg/min), and aerosol concentration (RAM-S; GCA Corp., Bedford, MA). Only one centrally located tier was used in each chamber to hold two, 16-unit (rat) modules to optimize aerosol distribution across all individual animal cages ($<12\%$ variation across 12 sampling sites). Actual aerosol concentration for each study exposure was determined each hour gravimetrically by collection of aerosol at 1.0 liters/min on type-A/E, glass-fiber filters (Gelman Sciences, Ann Arbor, MI) just above the holding cages. Once each exposure run, particle size distribution was determined using a seven-stage cascade impactor (Anderson, Atlanta, GA). Single, acute inhalation exposures (6 hr) to VDM were nominally 1000, 300, 100, 70, 40, and 10 mg/m³ with an additional exposure regime of 40 mg/m³, 6 hr/day for 5 days. The range of MMADs for the concentration-response studies was 3.9–5.4 μm . Actual exposure concentrations and particle size determinations for each of the studies are reported in Table 1. The 5-day exposure to 40 mg/m³ exhibited a daily mean ($\pm\text{SD}$) concentration of 38 ± 2 mg/m³ with a particle size distribution of 3.0 ± 2.1 μm (MMAD \pm σ).

Nose-only exposures were conducted with 20 rats/group restrained in whole-body/nose-protruding plexiglass tubes (sized for 250-g rats) placed inside the H-2000 chambers on top of the rat-holding cages and exposed to 300 mg/m³ (303 ± 33 mg/m³; MMAD 4.7 ± 2.0 μm) of VDM for 6 hr. This approach was taken to avoid or minimize the contamination of the rat fur with the VDM aerosol.

Lung instillation. Rats (18 males/group) were dosed via intratracheal instillation with 0.25 ml of the VDM as an aqueous suspension in saline. The low dose (250 μg) was chosen to be 10X greater than the burden of dye found in the lungs of rats immediately following 300 mg/m³ 6-hr inhalation exposure (~ 25 μg). The other two doses were 2X and 4X this low dose to minimize the chance of observing a toxic effect (500 and 1000 μg); saline sham served as the vehicle control. Time points chosen for observation and analysis were 1, 3, and 7 days postinstillation (6 rats/group). Prior to instillation, the syringe containing the dye suspension was vigorously shaken with a test tube vortex. A 0.5-ml bolus of air which was drawn into the syringe above the instillate fluid prior to vortexing to ensure complete delivery of the material. Previously, we have shown that particles can be reproducibly delivered ($93.8 \pm 2.0\%$ recovery) using this method (Costa *et al.*, 1986).

Gavage methods. The VDM and the two component dyes (DR11 and DB3) were each suspended in a 25% propylene glycol (PEG)/water solution. PEG was used to ensure dispersal of the particles within the aqueous vehicle and aid in dye absorption since administration would be in the form of a one-time bolus (2 ml) in contrast to the ~ 24 hr administration via preening. The gavage dose was 800 mg/kg for each of the component dyes and VDM. This dose was based on a calculated estimate of maximal body surface deposition occurring during whole-body exposure to 100 mg/m³ for 6 hr with subsequent ingestion by preening. The basic assumption for this calculation was that the entire body fur surface of the rat lay exposed cross-sectionally to the particles passing through the chamber cross-

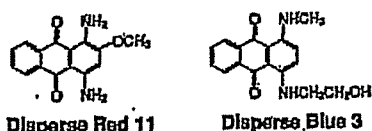


FIG. 1. Chemical structure of Disperse Red 11 (1,4-diamino-2-methoxy-anthraquinone) and Disperse Blue 3 (1-methylamino-4-hydroxyethylamino-anthraquinone).

TOXICITY OF AN ANTHRAQUINONE DYE MIXTURE

105

section at 15 air changes/hr for 6 hr. Time points of 1, 3, and 7 days postexposure were chosen for observation and analysis.

Tissue preparation. Rats were anesthetized with Nembutal (50 mg/kg). Serum was obtained by removing 5 ml of blood via the abdominal aorta. The blood was allowed to clot for 30 min and was then centrifuged. The serum was frozen at -20°C for subsequent determination of clinical chemistry endpoints. After exsanguination by cutting the abdominal aorta, the livers and lungs were removed and weighed, and a 0.25-g section from each was excised and homogenized in 3.0 ml of 6% metaphosphoric acid to precipitate tissue protein. The homogenates were centrifuged at 14,000g for 20 min at 4°C and the supernatants were decanted and stored at -80°C for determination of whole liver and lung non-protein-sulphydryl content (NPSH). The remaining liver and lung were homogenized in 50 mM Tris-HCl, 1.15% KCl, pH 7.4, with a weight-to-volume ratio of 1:3 and 1:7, respectively. The homogenates were centrifuged at 20,000g for 20 min at 4°C and the supernatants were decanted, recentrifuged at 100,000g for 1 hr at 4°C , and stored at -80°C for determination of glucose-6-phosphate dehydrogenase (G6PDH), glutathione reductase (GRD), glutathione peroxidase (GPX), glutathione S-transferase (GTR), and total protein. The microsomal pellets were resuspended in 50 mM Tris-HCl, 1.15% KCl, 25% glycerol, pH 7.4, and stored at -80°C for determination of NADPH cytochrome-c reductase (CYTOC) and cytochrome P450 (P450).

Clinical chemistry. All serum chemistries, alanine aminotransferase (ALT/SGPT), lactate dehydrogenase (LDH), sorbitol dehydrogenase (SDH), and isocitrate dehydrogenase (ICDH), were run on a Centrifichem System 500 centrifugal analyzer (Baker Instruments, Allentown, PA) using commercially prepared kits from Baker Instruments or Sigma Chemical Co. (St. Louis, MO).

Biochemical assays. The following biochemical assays were modified for liver and lung analysis on the Centrifichem System 500 centrifugal analyzer. The assay methods for total GPX and GRD have been reported elsewhere (Jaskot *et al.*, 1983). G6PDH assays were performed using 1.2 mM glucose 6-phosphate, 0.15 mM NADP, and 50 mM triethanolamine-HCl, pH 7.5, as modified from Mustafa *et al.* (1977). GTR assays were performed using 1-chloro-2,4-dinitrobenzene (CDNB) as substrate modified from Habig *et al.* (1974). The activity of CYTOC was assayed using horse heart cytochrome c as substrate as modified from Phillips and Langdon (1962). P450 was determined using a Cary 219 spectrophotometer by the method of Omura and Sato (1964). NPSH content was determined by a modification of the method of Sedlak and Lindsay (1968). The reagent contained 2.0 mM ethylenediaminetetraacetic acid and 0.21 mM 5,5'-dithiobis-2-nitrobenzoic acid (DTNB) in a 0.4 M Tris-HCl buffer, pH 8.9. Sample concentration of NPSH is determined from a standard curve. Protein concentration was determined using the Bio-Rad method (Bio-Rad Laboratories, Richmond, CA) for total protein determinations as modified for use on the centrifugal analyzer. The reagent contained a 1:5 dilution of the commercially prepared Bio-Rad dye reagent in deionized water and filtered prior to use. Sample protein concentration was determined from a standard curve using BSA standards.

Histopathology. All necropsies, histologic preparation of tissues, histopathologic evaluation of tissues, and photographs were provided by Experimental Pathology Labs, Inc., RTP, NC. Routine H + E-stained slides from paraffin-embedded tissues were prepared from the formalin-preserved nasal cavity (three regions), trachea, tracheobronchial lymph node, inflated lung, brain, stomach, duodenum, jejunum, ileum, cecum, colon, liver, spleen, and kidneys. All evaluations were done blind to the extent possible since most samples were tinted purple from the VDM exposures.

Lung burden and blood analysis. Lungs were removed from subgroups of the exposed rats ($n = 2$ to 6 rats/group as designated in Table 2), were dissected at the carina, and were homogenized in deionized water 1:4 (w/v) and frozen at -20°C until analysis. Frozen samples were thawed, diluted in 10 ml of DI water, and rehomogenized for 1 min (pulsed) using a Tekmar sonic disruptor. The samples were then extracted 3X with 10 ml of methylene chloride with each emulsion diluted to 50 ml and finally con-

centrated to 1 ml by evaporation under N_2 . Each sample was diluted to 2 ml in acetonitrile and filtered through an Alltech PTFE (250 mm \times 0.45- μm) syringe filter prior to HPLC analysis. The following conditions for HPLC analysis of the VDM were used: Isocratic 95/5 acetonitrile/methylene chloride mobile phase with a flow rate of 1 ml/min; Whatman PAC column (250 mm \times 5 mm i.d., 10- μm particle size), 254 nm uv detection, a 40- μl sample injection volume. Under these conditions it was possible to separate the sample components and to quantitate the peak containing the coeluting DR11 and DB3 components by manual integration. The sample values were then adjusted by recovery values (57.9%) previously determined from rat lung samples spiked with varying concentrations of the dyes. Analysis of selected blood samples for VDM was conducted similarly, but with blood diluted in water rather than lung tissue. Attempts to extract the VDM from urine were abandoned when methylene chloride failed, indicating that the colored compound was a water-soluble metabolite and we were not prepared to analyze it.

Statistical analysis. The data were analyzed using a two-way analysis of variance. The two factors were treatment (at various levels of exposure) and time postexposure (days). Significant interaction resulted in subtesting among levels of the variables examining the standardized differences in the least-square means. A p value of less than or equal to 0.01 indicated statistical significance due to multiple comparisons.

RESULTS

Inhalation

There was a trend for particle size to correlate with chamber aerosol concentration (i.e., particle size increased with concentration, Table 1). The reasons for this were likely due to the higher feed rate of the jet mill required to achieve the highest concentrations (300 and 1000 mg/m^3). This would result in less impact time for disruption of particle agglomerates during sonic dispersion as well as increase the chance of reagglomeration of these self-adherent particles in the inlet conduit to the chamber. However, at VDM levels of 100 mg/m^3 and less, particle sizes were stabilized.

Table 2 summarizes the results from the inhalation exposures with VDM. Immediately after the single 6-hr whole-body exposures, all rats showed fur deposition of VDM. As a general observation, the degree of coloration of the fur was concentration-dependent, with all rats exhibiting vigorous and persistent preening activity. By morning, the ani-

TABLE 1
Aerosol Exposure Characteristics

| Nominal VDM concentration (mg/m^3) | Actual VDM concentration ^a (mg/m^3) | Particle size ^b (μm) |
|--|--|--|
| 1000 | 933 \pm 38 | 5.4 \pm 2.1 |
| 300 | 321 \pm 25 | 4.6 \pm 2.1 |
| 100 | 105 \pm 27 | 4.0 \pm 2.3 |
| 70 | 71 \pm 8 | 3.9 \pm 2.1 |
| 40 | 43 \pm 4 | 3.9 \pm 2.2 |
| 10 | 13 \pm 2 | 3.9 \pm 2.1 |

^a Means \pm SD.

^b MMAD \pm σ .

106

JASKOT AND COSTA

TABLE 2
Summary: Inhalation Exposures

| Violet dye mix (mg/m ³) ^a | Postexposure recovery | Histopathology | VDM lung burdens (µg/g lung) |
|--|--|--|---|
| 1000 | All rats (18) died by Day 3 postexposure. | ND | Day 0, 57 ^d Day 3, 78 ^d |
| 300 | 9/18 rats died by Day 3 postexposure. The remaining rats were moribund and hence killed. | Liver: Severe centrilobular degeneration and necrosis. Nasal: Degeneration and necrosis of olfactory epithelium. | Day 0, 25.5 ± 1.6 ^e Blood, 4.61 ^f Day 3, 18.9 ± 2.1 ^e Blood, 2.09 ^f (moribund or dead rats) |
| 100 | All rats lived (30) through the 3- and 7-day holding period. | Liver: (Day 3) Moderate degeneration and necrosis. (Day 7) Normal. Nasal: (Day 3) Degeneration, necrosis, and sloughing of olfactory epithelium. (Day 7) Slight degeneration present. | Day 0, 6.50 ± 1.03 ^e Blood, 2.2 ± 0.26 ^f Day 3, 0.90 ± 0.57 ^e Blood, 0.2 ± 0.2 ^f Day 7, 0.03 ± 0.01 ^e Blood, <0.03 ^f |
| 70 | All rats (18) lived through the 3- and 7-day holding period. | Liver: Normal. Nasal: (Day 3) Moderate degeneration and necrosis of olfactory epithelium. (Day 7) Slight degeneration. | Day 0, 3.00 ^e Day 3, 0.30 ^e Day 7, <0.03 |
| 40 | All rats (18) lived through the 3- and 7-day holding period. | Liver: Normal. Nasal: (Day 3) Moderate degeneration and necrosis of olfactory epithelium. (Day 7) Minimal degeneration. | Day 0, 6.40 ^e Day 3, 0.08 ^e Day 7, 0.03 ^e |
| 10 | 16/18 rats lived through the 3- and 7-day holding period. | Liver: Normal. Nasal: (Day 3) Slight degeneration and necrosis of olfactory epithelium. (Day 7) Minimal degeneration. | Day 0, 1.40 ^e Day 3, <0.03 ^e Day 7, <0.03 ^e |
| 40 ^b | 16/32 rats were dead or moribund by Day 2 postexposure. The remaining rats survived to Day 7 postexposure. | ND | ND |
| 300 ^c | 18/20 rats lived through the 3- and 7-day holding period. | Liver: Normal. Nasal: (Day 3) Degeneration, necrosis, and sloughing of olfactory epithelium. (Day 7) Slight degeneration present. | Day 0, 64.5 ± 16.4 ^e Day 3, ND Day 7, ND |

Note. ND, no data.

^a Whole-body 6-hr exposure unless otherwise noted.^b Whole-body 5-day exposure, 6 hr/day.^c Nose-only 6-hr exposure.^d 2-3 rats.^e 6 rats.^f µg/ml whole blood.

mals were virtually clean with only a stripe of violet coloring on the cranial-dorsum area, presumably an area out of reach of the animals' preening strokes. Violet-colored fecal material was evident as was violet-colored urine. Rats exposed to the highest VDM concentrations (1000 and 300 mg/m³) consistently began to deteriorate during Day 2 with most deaths occurring between Days 2 and 3. By Day 3, the mortality incidence at 1000 mg/m³ was 18 dead/18 and at

300 mg/m³ 9 dead + 9 moribund/18. At lower concentrations, the violet coloring was qualitatively similar, but quantitatively less (by observation), with the physical condition of the rats not visibly affected.

Tissues of the moribund animals (*n* = 5; 300 mg/m³ group) were prepared for histopathologic evaluation. Only severe centrilobular liver damage and necrosis of the nasal olfactory epithelium were apparent in the exposed animals

TOXICITY OF AN ANTHRAQUINONE DYE MIXTURE

107

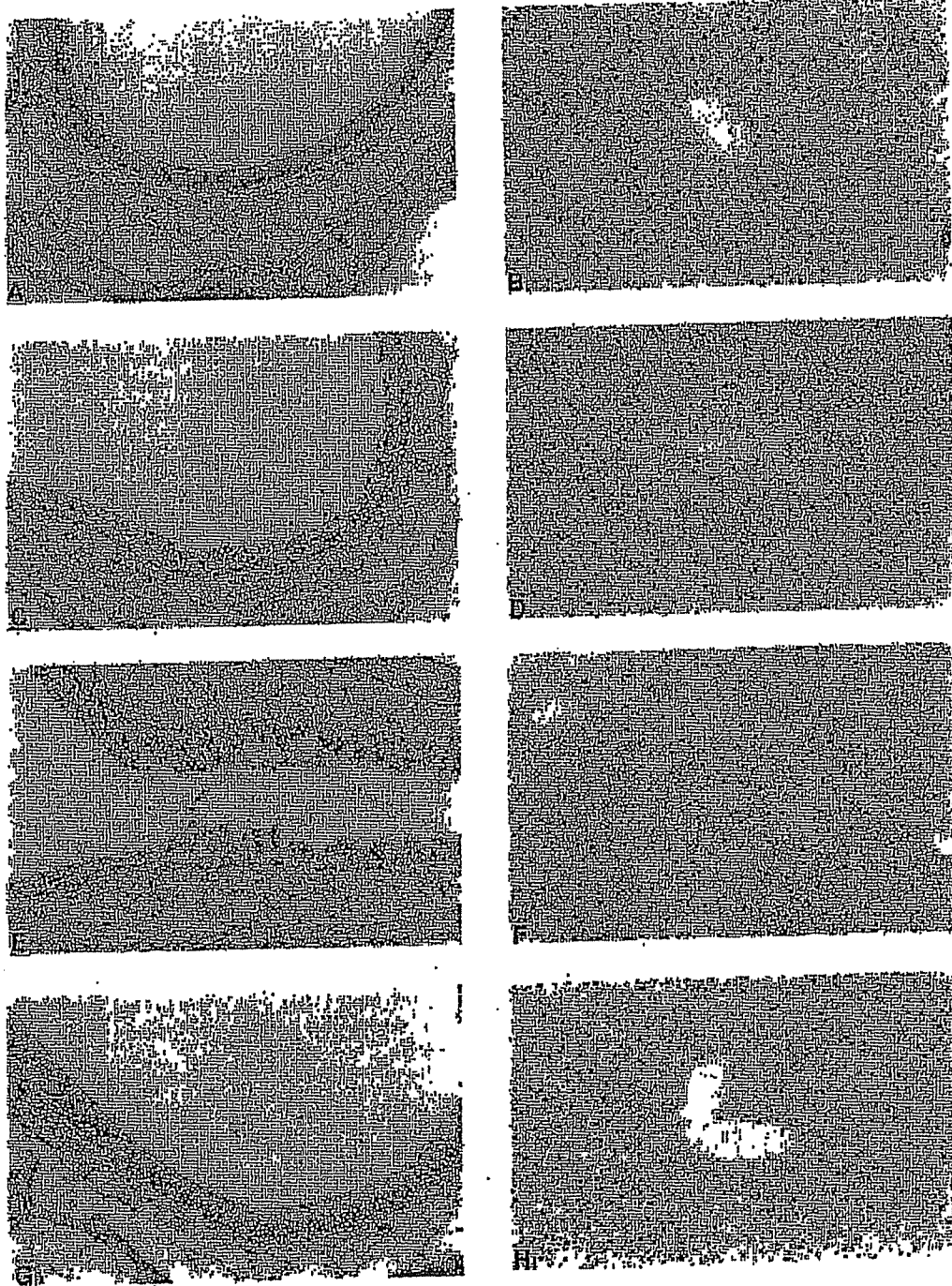


FIG. 2. Olfactory epithelium (left photos) and liver centrilobular region (right photos) following inhalation exposure to air or VDM. Air (A, B), 100 mg/m³ VDM for 6 hr, 3 days postexposure (C, D), 300 mg/m³ VDM for 6 hr, 8 days postexposure (E, F), 100 mg/m³ VDM for 6 hr, 7 days postexposure (G, H). All photos are magnified 250%, except E which is 400%.

(Fig. 2). Pilot assessments of serum SGPT and LDH from the moribund animals indicated 50X and 10X increases, respectively, for these liver injury enzymes as compared to control. Other tissues, including the lung airway and parenchyma, appeared normal. Analogously exposed rats (100, 70, and 40 mg/m³, Table 2) did not experience mortality up to 7 days postexposure. However, 2 rats/18 in the 10 mg/m³ group did succumb, somewhat surprisingly, between Days 2 and 3; their tissues were not assessed due to rigor. The tissues affected in the 100 mg/m³ animals were the same as those of the higher concentrations. Centrilobular liver damage at Day 3 was somewhat moderated and largely regressed to normal by Day 7 (Fig. 2). Pilot serum chemistry data from these same animals revealed correspondingly elevated SGPT and LDH levels (29X and 2X, respectively) at Day 3 also returning to normal by Day 7. Similarly, the nasal olfactory epithelium appeared degenerated and necrotic at 3 days, but, unlike the liver, it was not fully restored at Day 7 (Fig. 2).

Exposures at 70, 40, and 10 mg/m³ resulted in no apparent hepatocellular pathology at the light microscopic level. However, in contrast, these rats clearly exhibited concentration-dependent, moderate to slight nasal olfactory cell degeneration. These lesions waned considerably by Day 7 postexposure, but evidence of damage persisted, mostly in the form of incompletely restored architecture of the olfactory epithelium.

Eighteen of 20 rats exposed "nose-only" to 300 mg/m³ survived through 7 days postexposure, in contrast to the whole-body studies. The 2 animals that died were visibly contaminated with VDM by the end of exposure due to failure of the nose seal. Histologic evaluation of the livers of rats killed at Day 3 was unremarkable, while the nasal olfactory epithelium showed degeneration, with characteristic sloughing and necrosis. The damage was extensive, essentially as that seen previously with the whole-body exposures. At Day 7 postexposure, a slight degeneration of the olfactory epithelium again persisted; the livers, however, remained normal.

Fifty percent of the rats exposed to 40 mg/m³ for 6 hr/day for 5 days were dead or moribund by Day 2 after the 5-day exposure regime. The moribund rats that were killed for evaluation at Day 3 had serum SGPT and LDH levels 5–10 times above control rats. By Day 7 postexposure, the surviving rats had normal serum enzyme levels.

Lung burdens were determined in subgroups of rats from each exposure level ($n = 2 - 6$) at 0, 3, and 7 days after exposure (Table 2). Lung weights were not affected by the VDM exposures. The lungs of rats exposed to 1000 mg/m³ ($n = 2$) contained 57 μg VDM/g of tissue immediately post-exposure. Assay of two dead rats (prior to rigor) on Day 3 indicated that little if any clearance from the lung had occurred (78 μg VDM/g). Although this latter value may have been influenced by the fact that the assay was conducted in dead animals, analogous results were obtained in rats ex-

posed to 300 mg/m³. Immediately after exposure, these lungs contained 25.5 ± 1.65 μg VDM/g ($n = 6$) as compared to 18.9 ± 2.1 μg VDM/g at 3 days ($n = 6$ live but moribund animals). Interestingly, in the nose-only exposed animals ($n = 6$), the 0-time lung burden was 64.5 ± 16.6 μg VDM/g of lung tissue, ~ 2.5 X the dose of the whole-body exposed lungs (as noted above, no liver, but clear olfactory tissue damage was observed).

At exposures ≤ 100 mg/m³, more than 85% of the initial lung burden was cleared by Day 3; virtually all the lung deposited VDM was cleared by Day 7 ($n = 6$ for each time point). The VDM retention in the 100 mg/m³ group at Day 0 was 6.5 ± 1.0 μg /g, about 26% of the 300 mg/m³ group. The variation among the other exposure groups at the Day 0 time point probably reflects the small sample sizes ($n = 2$), although the between-animal variability in each group was not particularly variant ($<40\%$).

Lung Instillation

Serum LDH, SGPT/ALT, SDH, and ICDH were not significantly different from control at any VDM concentration. Also, lung and liver G6PDH, GRD, GTR, GPX, NPSH, CYTOC, and P450 were not significantly different from control at any VDM concentration or time postinstillation.

Gavage

The results of serum enzymes of liver injury from rats gavaged with 800 mg/kg of DB3, DR11, or VDM are illustrated in Fig. 3. The DB3 component of the VDM caused a slight but significant elevation in all of the enzymes (3–6X control) by Day 3 post-treatment. By Day 7, these were back to control levels. The DR11 component caused minimal enzyme changes (-1.5 to 2.5 X) that were rarely significantly different (exceptions: Day 1 LDH; Day 7 SGPT) from control up to 7 days. The VDM mix, however, caused substantial increases (10- to 100-fold) in SGPT, SDH, and ICDH at both 1 and 3 days postgavage. By Day 7, all enzyme values were near control values. No mortality was observed.

Liver G6PDH and GRD were significantly elevated 1, 3, and 7 days postgavage for all three dyes with the exception of GRD, which at Day 1 was no different from control following gavage with VDM (Fig. 4). Liver GPX was significantly increased on Days 1 and 3 following gavage with DB3 and DR11 and at Day 7 postgavage with DR11. NPSH was significantly increased on Day 3 postgavage with DB3 and Day 7 postgavage with DB3 and VDM (Fig. 4).

As depicted in Fig. 5, liver GTR was significantly elevated on Days 1, 3, and 7 postgavage with DB3 and DR11, while no change from control was seen at Days 1 and 7 postgavage with VDM. In contrast, a significant decrease in

TOXICITY OF AN ANTHRAQUINONE DYE MIXTURE

109

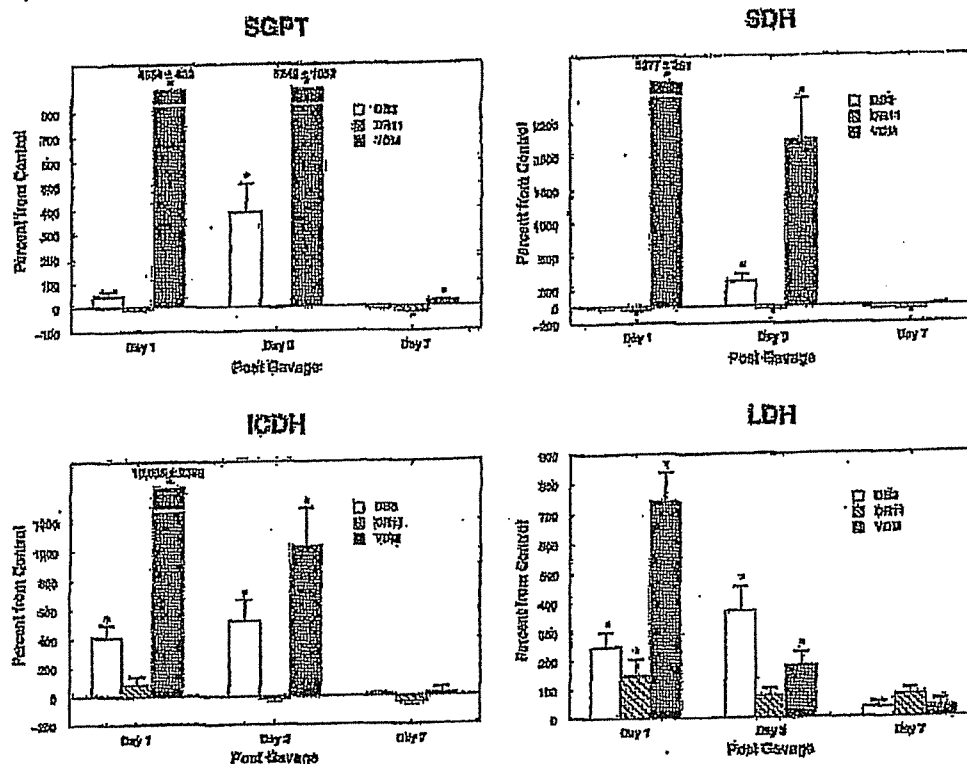


FIG. 3. Percentage from control of serum enzymes 1, 3, and 7 days after gavage with 800 mg/kg of DB3, DB11, and VDM (means \pm SE, $n = 6$). *Statistically different from control at $p < 0.05$.

GTR was observed at Day 3. The significant increases in CYTOC were observed at Days 1 and 3 postgavage with DB3 and DB11 (50 to 130%) contrasts with the significant decreases (-25 to -50%) in CYTOC due to VDM at Days 3 and 7 postgavage and at Day 7 postgavage with DB3 (Fig. 5). Liver P450 was significantly elevated Day 1 postgavage with DB3 and DB11 and Day 3 postgavage with DB11, but was significantly decreased at Day 3 postgavage with VDM and Day 7 postgavage with DB3 and VDM (Fig. 5).

DISCUSSION

This study evaluated the toxicity of a mixture of two anthraquinone dyes (92.9% DB11 [1,4-diamino-2-anthoxy-anthraquinone]; 7.1% DB3 [1-methylamino-4-hydroxyethylamino-anthraquinone]) in rats following a single 6-hr inhalation exposure. In its formulation, this anthraquinone mixture was formed from solubilization of the red and blue dyes and spray-dried to a fine, tenacious violet powder. Aerosol generation for exposure to rats resulted in particles in the 3- to 5 μ m range, a particle size range which

led to significant surface deposition as well as lung and nasal retention. The pattern of results in the inhalation exposure studies suggested that the systemic toxicity observations were probably not due to inhalation of the dust per se (lung deposition), but rather to the oral ingestion (via preening) of the dye adhering to the fur of the animals. Toxicity was limited to the liver and olfactory epithelial tissues of the nose while the lung itself and the 12 other organ tissues examined appeared to have been spared. Follow-up experiments were conducted utilizing direct intratracheal instillation of the VDM, as well as gavage of the VDM and its constituent dyes to better characterize the nature of the toxicity. It appears that a significant systemic dose of VDM could be achieved via preening behavior of the animals, leading to gut absorption and liver toxicity. The studies with the constituent dyes suggested a likely metabolic interaction between these dyes, with the resultant toxicity being much greater than either dye alone or their additive sum. What specific metabolism was involved is uncertain (apparently unique—in kind or degree—to the liver and, curiously, the olfactory epithelium). The observed nasal olfactory toxicity, itself, was probably the re-

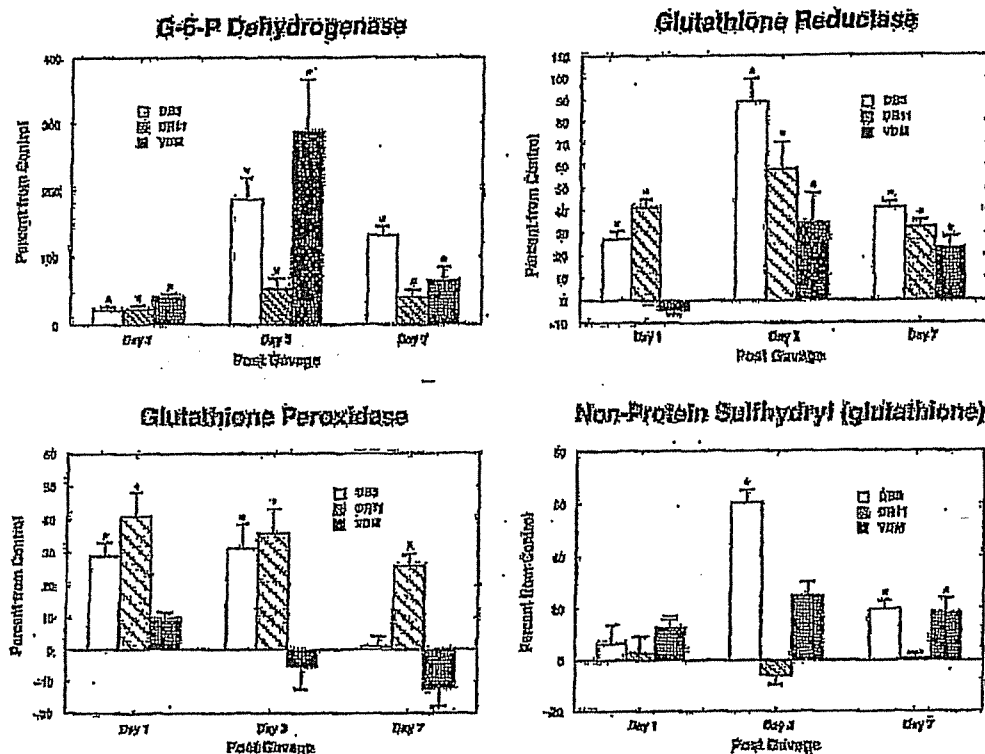


FIG. 4. Percentage from control of liver glutathione-related enzymes 1, 3, and 7 days after exposure with 300 mg/kg of DBS, DBS1, and VDM means \pm SE, $n = 6$. *Statistically different from control at $p < 0.01$.

sult of direct nasal deposition of VDM during inhalation and not redistribution of other toxic entities.

Whole-body inhalation exposure of male and female rats to high concentrations of VDM (1000 or 300 mg/m³) was essentially lethal within 3 days postexposure. Death appeared to be due to severe liver necrosis. Lower concentrations also resulted in concentration-dependent liver damage by Day 3 which largely resolved by Day 7. Surprisingly, no pathology was apparent in the lungs of rats at any exposure concentration despite deep lung retention of substantial amounts of dye. Respiratory tract lesions were isolated to the nose where deposition of these large dye particles ($\sim 4 \mu\text{m}$ MMAD) would be expected to be high. Deposition of the dye on the fur of exposed rats was also very high (subjectively appearing to be concentration-dependent), which as is typical was followed by vigorous preening by the animals. When rats were exposed nose-only to 300 mg/m³ for 6 hr (a lethal whole-body exposure), no mortality or liver injury was observed except in two animals which broke their nose seals and had visibly contaminated upper body areas. Again, no lung pathology was apparent despite the $\sim 3\times$ greater lung burden in these nose-only exposed

rats (7825 μg). Indeed, no evidence of lung toxicity was apparent with lung instillation of up to 1000 μg , almost $1.5\times$ the dose determined in the lungs of these nose-only exposed animals.

The localization of the respiratory tract pathology to the nasal olfactory epithelium, seen even at 10 mg/m³, and complete absence of other respiratory damage suggest that these cells were, for some reason, unusually sensitive to the VDM. Several investigators have shown that nasal epithelium contains biotransformation enzymes capable of metabolizing various organic compounds (Boyd, 1983; Foster *et al.*, 1986; and Hadley and Dahl, 1982). One explanation for the degeneration and necrosis solely of the olfactory epithelium would involve a specific unusual susceptibility via a critical target attacked by the VDM chemical mix. Perhaps a more credible hypothesis would involve metabolism of the VDM to a toxic metabolite which acts immediately and does not diffuse to neighboring cells (respiratory epithelium) within the nose, which remain unaffected because they lack that specific metabolic activity. The cellular demarcation of toxicity was clear (Fig. 2), despite the fact that the nasal deposition of the VDM particles would certainly

TOXICITY OF AN ANTHRAQUINONE DYE MIXTURE

111

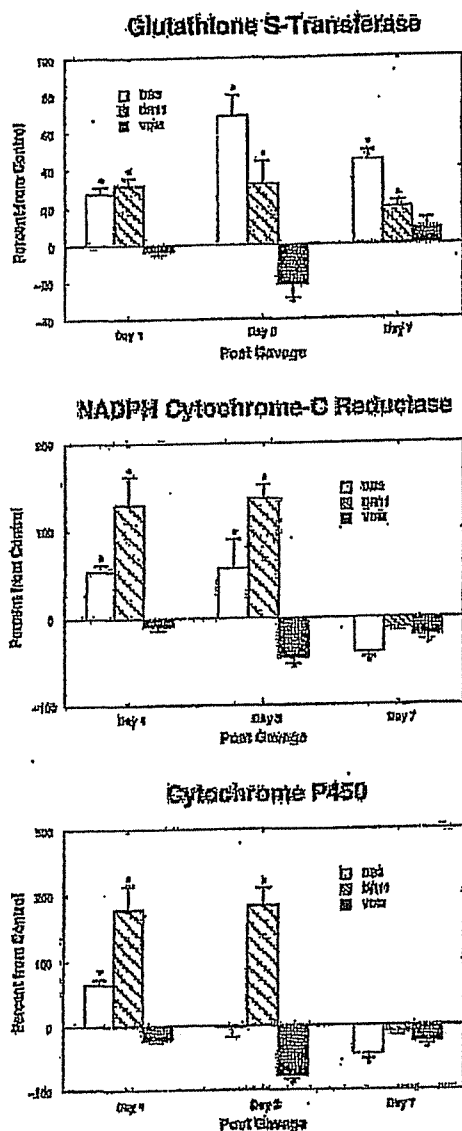


FIG. 5. Percentage from control of liver xenobiotic enzymes 1, 3, and 7 days after gavage with 800 mg/kg of DB3, DB11, and VDM (means \pm SE, $n = 6$). *Statistically different from control at $p < 0.01$.

have breached the separation between cell types. Because the olfactory and the liver lesions paralleled each other (particularly at the high concentration exposures when dose to the liver was likely substantial), a similarity in the toxic mechanism might be suggested, although we have no direct evidence in support of this hypothesis. Moreover, the presence of olfactory damage at inhaled VDM concentrations

which resulted in no liver injury would support the contention that the olfactory cells are not simply being affected by a circulating metabolite originating in the liver.

Anthraquinone dyes deposited within the lung, because of their relatively low lipid and water solubilities, would probably not be readily transported across the air-blood interface into the blood. The particles would only slowly dissolve in the lung fluids, and would thus more likely be available for macrophage/mucociliary clearance. While not assessed directly, the clearance of VDM was consistent with this scenario. Moreover, those animals suffering acute, lethal systemic toxicity appeared to clear little or none of the deposited VDM, probably due to the poor health condition of the animals. Gut transport of food (and dye) continued immediately postexposure appeared also to be largely inhibited. On the other hand, the lung burdens in animals less affected systemically (100 mg/m³) were readily cleared (>85% by Day 3) from the lungs. Blood concentrations of dye were generally low (~ 2 μ g/ml; Table 2), but if adjusted for total blood volume (~ 10 ml), the circulating extractable VDM would exceed the original dose retained by the lung. The violet coloring of the serum and urine appeared to be a solubilized form of VDM (i.e., metabolite) since it was not extractable with methylene chloride. It seems unlikely that mucociliary clearance and swallowing (or even dissolution) of the lung-retained dye could have added significantly to the systemic dose. This was confirmed by the lung instillation studies which revealed no local pulmonary toxicity or systemic effects.

We also exposed rats for 5 consecutive days at 6 hr/day to a low concentration of VDM (40 mg/m³) that had previously shown limited toxic effects after one 6-hr exposure to address the question of whether tolerance to the toxic effect of VDM would occur or whether toxicity would be cumulative. Although the rats did not show untoward effects during the 5-day exposure, 2 days after its completion, 50% (16/32) of the rats had died. These findings lead us to conclude that the VDM toxicity observed was cumulative due to repeated daily ingestion of dye from preening. As a simple assessment, the dose accumulated, if assumed to be linear by daily preening with minimal fecal loss, would fall between that resulting from the 100 mg/m³ (no mortality and moderate liver injury) and 100 mg/m³ (lethal with severe liver necrosis) exposures.

In the gavage studies with VDM and its constituent dyes (DB3 and DB11), the DB11 component appeared to have negligible liver toxicity (as per serum chemistry) at a dose about that which may have been ingested as VDM during the inhalation exposure, while the DB3 component was only slightly toxic at ~ 40 times that relative VDM dose. In contrast, VDM itself caused the rapid onset of liver toxicity 1 day following gavage with a hepatotoxicity pattern analogous to that derived from the whole-body inhalation exposure. However, the intensity of effect in terms of serum enzyme responses in the gavaged rats versus "preening

rats" occurred earlier (Day 1, SGPT 47X:1X; LDH 8X:1X) and was higher (Day 3, SGPT 52X:19X; LDH 3X:1X), which may have reflected dosimetric differences relative to the preened dose or dose rate or perhaps the influence of the 25% PEG/aqueous vehicle used in the gavage study. Interestingly, there was no mortality among the gavaged animals although the SGPT and LDH value of these animals approximated those of rats moribund 3 days after 300 mg/m³. Unfortunately, the lack of histopathological evaluation in the gavaged animals makes uncertain the degree of actual histologic damage in these animals.

These data suggest that a synergistic interaction occurred between the two component dyes (DR11 and DB3) in VDM to elicit the observed toxicity in the liver and olfactory epithelium of exposed rats. Since metabolism was not specifically analyzed in this study, suggested mechanisms remain inferential and therefore speculative. We hypothesize that liver (and olfactory epithelial) cells elaborate a toxic metabolite from the DB3 component by a mechanism which is potentiated via the DR11-induced activity of the P450 (observed to increase by more than 200% as early as 24 hr post-treatment). While an alternative explanation might include the inhibition of a detoxification step, we believe the lack of direct toxic effect of DR11 on the liver itself and similar, although much reduced, toxicity of DB3 as compared to VDM at ~40X the constituent dose, would better support the contention of potentiation. Clearly, direct study of this interaction would be needed to elucidate this issue. Why only olfactory and liver cells are affected remains unclear and may be due to a unique enzyme or P450 isoenzyme found only in these tissues.

In conclusion, the anthraquinone dyes, DR11 and DB3, appear to be minimally toxic when individually administered to rats. However, when combined, they are capable of interacting, perhaps via P450-related metabolic pathways, to specifically damage centilobular hepatocytes and olfactory epithelial cells. The lung itself, which in humans would be a primary portal of entry of VDM under occupational or field conditions, was unaffected by VDM in rats even at very high instilled doses. Exposures to VDM via the digestive tract, however, unveil significant potential for toxicity. This phenomenon reinforces the need to consider realistic exposure situations, including the portals of entry and the exposure mixtures, in the assessment of toxic risks.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the technical assistance Inhalation Exposure Group of the Pulmonary Toxicology Branch, particularly the Mantech, Inc. staff of Mr. M. Higuchi, Mr. J. Sandy, and Mr. D. Terrell for the inhalation exposures and characterizations, Ms. J. Richards for serum and liver biochemistry measurements, and Drs. R. Zweifinger and S.

Cooper for chemical assay of tissue dye burdens. Histopathological assessments were performed by Dr. William F. MacKenzie of Experimental Pathology Laboratory, Inc.

REFERENCES

- Bond, J. A. (1983). Some biotransformation enzymes responsible for polycyclic aromatic hydrocarbon metabolism in rat nasal turbinates: Effects on enzyme activities of *in vitro* modifiers and inhalation exposure of rat to inducing agents. *Cancer Res.* 43, 4803-4811.
- Costa, D. L., Lehmann, J. R., Harold, W. M., and Drew, R. T. (1986). Transoral tracheal intubation of rodents using a fiberoptic laryngoscope. *Lab Anim. Sci.* 36(3), 256-261.
- Foster, J. R., Elcombe, C. R., Boobis, A. R., Davies, D. S., Sesardic, D., McQuade, J., Robson, R. T., Hayward, C., and Lock E. A. (1986). Immunocytochemical localization of cytochrome P-450 in hepatic and extra-hepatic tissues of the rat with a monoclonal antibody against cytochrome P-450 c. *Biochem. Pharmacol.* 35, 4543-4554.
- Habig, W. H., Pabst, M. J., and Jakoby, W. B. (1974). Glutathione S-transferases: The first enzymatic step in mercapturic acid formation. *J. Biol. Chem.* 249, 7130-7139.
- Hadley, W. M., and Dahl, A. R. (1982). Cytochrome P-450 dependent monooxygenase activity in rat nasal epithelial membranes. *Toxicol. Lett.* 10, 417-422.
- Hatch, K. L. (1984). Chemicals and textiles. I. Dermatological problems related of fiber content and dyes. *Textile Res. J.* 54, 664-682.
- Higuchi, M. A., and Davies, D. W. (1990). *Inhalation Toxicology of Red and Violet Dye Mixtures: Chamber Concentration and Particle Distribution Report*. U.S. Army Medical Research and Development Command, AD-TR-90-A211-266.
- Higuchi, M. A., and Steinhagen, W. H. (1991). Modification and characterization of dry material feeder delivery of red and violet dye mixtures. *Inhalation Toxicol.* 3, 223-235.
- Jaskot, R. H., Charlet, E. G., Gross, E. C., and Grady, M. A. (1983). An automated analysis of glutathione peroxidase, S-transferase, and reductase activity in animal tissue. *J. Anal. Toxicol.* 7, 86-88.
- Mustafa, M. G., Hacker, A. D., Ospital, J. J., Hussain, M. Z., and Lee, S. D. (1977). Biochemical effects of environmental oxidant pollutants in animal lungs. In *Biochemical Effects of Environmental Pollutants* (S. D. Lee, Ed.), pp. 59-96. Ann Arbor Science, Ann Arbor, MI.
- NIOSH (1981). *Anthraquinone Dye: Toxicological Profiles*. Final Report, US Consumer Product Safety Commission, Contract No. CPSC-C-81-1110, Washington, DC.
- Omura, T., and Sato, R. (1964). The Carbon monoxide-binding pigment of liver microsomes. II. Solubilization, purification, and properties. *J. Biol. Chem.* 239, 2379-2385.
- Phillips, A. H., and Langdon, R. G. (1962). Hepatic triphosphopyridine nucleotide-Cytochrome C reductase: Isolation, characterization, and kinetic studies. *J. Biol. Chem.* 237, 2652-2660.
- Sedlak, J., and Lindsay, R. H. (1968). Estimation of total, protein-bound, and nonprotein sulfhydryl groups in tissue with ellman's reagent. *Anal. Biochem.* 25, 192-205.
- Sendelbach, L. E. (1989). A review of the toxicity and carcinogenicity of anthraquinone derivatives. *Toxicology* 57, 227-240.
- Sun, J. D., Henderson, R. F., Marshall, T. C., Cheng, Y.-S., Dutcher, J. S., Pickrell, J. A., Mauderly, J. L., Hahn, F. F., Baas, D. A., Seiler, F. A., and Hobbs, C. H. (1987). The inhalation toxicity of two commercial dyes: Solvent yellow 33 and solvent green 3. *Fundam. Appl. Toxicol.* 8, 358-371.

Docket No.: 40678REX(70329)
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT: James W. Thackeray et al.

Application No.: 90/008,359

Confirmation No.: 8757

Filed: December 4, 2006

Art Unit: 3991

For: ANTIHALATION COMPOSITIONS

Examiner: S. J. Stein

INFORMATION DISCLOSURE STATEMENT (IDS)

MS Ex Parte Reexam
ATTN: Central Reexamination Unit
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

Pursuant to 37 CFR 1.56, 1.97 and 1.98, the attention of the Patent and Trademark Office is hereby directed to the references listed on the attached PTO/SB/08. It is respectfully requested that the information be expressly considered during the prosecution of this application, and that the references be made of record therein and appear among the "References Cited" on any patent to issue therefrom.

This Information Disclosure Statement is submitted after the mailing date of the first Office Action on the merits, but before the mailing date of a Final Office Action or Notice of Allowance (37 CFR 1.97(c)).

Applicants submit herewith a copy of an Office communication issued in cited U.S. application number 90/008,360.

In accordance with 37 CFR 1.97(g), the filing of this Information Disclosure Statement shall not be construed to mean that a search has been made or that no other material information as defined in 37 CFR 1.56(a) exists. In accordance with 37 CFR

Application No.: 90/008,359

2

Docket No.: 40678REX(70329)

1.97(h), the filing of this Information Disclosure Statement shall not be construed to be an admission that any patent, publication or other information referred to therein is "prior art" for this invention unless specifically designated as such.

It is submitted that the Information Disclosure Statement is in compliance with 37 CFR 1.98 and the Examiner is respectfully requested to consider the listed references.

Please charge our Deposit Account No. 04-1105 in the amount of \$310.00 covering the fee set forth in 37 CFR 1.17(p), 1.20(d). The Director is hereby authorized to charge any deficiency in the fees filed, asserted to be filed or which should have been filed herewith (or with any paper hereafter filed in this application by this firm) to our Deposit Account No. 04-1105, under Order No. 40678REX(70329). A duplicate copy of this paper is enclosed.

Dated: June 11, 2007

Respectfully submitted,

By 

Peter F. Corless

Registration No.: 33,860

EDWARDS ANGELL PALMER & DODGE
LLP

P.O. Box 55874

Boston, Massachusetts 02205

(617) 517-5557

Attorneys/Agents For Applicant

PTO/SB/08A/B (09-06)

Approved for use through 03/31/2007. OMB 0661-0031

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|---|--------------------------|--------------------|
| Substitute for form 1449/PTO | | Complete if Known | |
| | | Application Number | 90/008,359 |
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Use as many sheets as necessary) | | Filing Date | December 4, 2006 |
| | | First Named Inventor | James W. Thackeray |
| | | Art Unit | 3991 |
| | | Examiner Name | S. J. Stein |
| Sheet | 1 | of | 1 |
| | | Attorney Docket Number | 40678REX(70329) |

| U.S. PATENT DOCUMENTS | | | | | |
|-----------------------|--------------------------|--|--------------------------------|--|---|
| Examiner Initials* | Cite No. ¹ | Document Number | Publication Date MM-DD-YYYY | Name of Patentee or Applicant of Cited Document | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear |
| | | Number-Kind Code ² (if known) | | | |
| | AA* | US-2006/0110882 | 05-25-2006 | Thackeray et al. | |
| | AB | US-90/008,360 | | Thackeray et al. | |

| FOREIGN PATENT DOCUMENTS | | | | | |
|--------------------------|--------------------------|---|-----------------------------------|--|---|
| Examiner Initials* | Cite No. ¹ | Foreign Patent Document | Publication Date MM-DD-YYYY | Name of Patentee or Applicant of Cited Document | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear |
| | | Country Code ³ -Number ⁴ -Kind Code ⁵ (if known) | | | |
| | | | | | |

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 608. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. * CITE NO.: Those application(s) which are marked with an single asterisk (*) next to the Cite No. are not supplied (under 37 CFR 1.98(a)(2)(iii)) because that application was filed after June 30, 2003 or is available in the IFW. ¹ Applicant's unique citation designation number (optional). ² See Kinds Codes of USPTO Patent Documents at www.uspto.gov or MPEP 801.04. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

| NON PATENT LITERATURE DOCUMENTS | | | |
|---------------------------------|--------------------------|---|----------------|
| Examiner Initials | Cite No. ¹ | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published. | T ² |
| | | | |
| | | | |

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 608. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ Applicant's unique citation designation number (optional). ² Applicant is to place a check mark here if English language Translation is attached.

| | | | |
|-----------------------|--|--------------------|--|
| Examiner Signature | | Date Considered | |
|-----------------------|--|--------------------|--|



UNITED STATES PATENT AND TRADEMARK OFFICE

Commissioner for Patents
United States Patent and Trademark Office
P.O. Box 1450
Alexandria, VA 22313-1450
www.uspto.gov

3/23/07

THIRD PARTY REQUESTER'S CORRESPONDENCE ADDRESS

CONSTANCE S. HUTTNER
SKADDEN ARPS SLATE MEAGHER & FLOM LLP
FOUR TIMES SQUARE
NEW YORK NY 10036

EX PARTE REEXAMINATION COMMUNICATION TRANSMITTAL FORM

REEXAMINATION CONTROL NO 90/008360
PATENT NO. 6,773,864
ART UNI 3992

Enclosed is a copy of the latest communication from the United States Patent and Trademark Office in the above identified ex parte reexamination proceeding (37 CFR 1.550(f)).

Where this copy is supplied after the reply by requester, 37 CFR 1.535, or the time for filing a reply has passed, no submission on behalf of the ex parte reexamination requester will be acknowledged or considered (37 CFR 1.550(g)).



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
 United States Patent and Trademark Office
 Address: COMMISSIONER FOR PATENTS
 P.O. Box 1450
 Alexandria, Virginia 22313-1450
 www.uspto.gov

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|-------------|----------------------|---------------------|------------------|
| 90/008,360 | 01/19/2007 | 6773864 | | 8758 |
| 21874 | 7590 | 03/23/2007 | EXAMINER | |
| EDWARDS & ANGELL, LLP P.O. BOX 55874 BOSTON, MA 02205 | | | ART UNIT | PAPER NUMBER |

DATE MAILED: 03/23/2007

Please find below and/or attached an Office communication concerning this application or proceeding.

| | | | |
|--|---------------------------|---------------------------------------|--|
| Order Granting / Denying Request For Ex Parte Reexamination | Control No. 90/008,360 | Patent Under Reexamination 6773864 | |
| | Examiner Alan Diamond | Art Unit 3991 | |

--The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

The request for *ex parte* reexamination filed 19 January 2007 has been considered and a determination has been made. An identification of the claims, the references relied upon, and the rationale supporting the determination are attached.

Attachments: a) ☐ PTO-892, b) ☒ PTO/SB/08, c) ☐ Other: _____

1. ☒ The request for *ex parte* reexamination is GRANTED.

RESPONSE TIMES ARE SET AS FOLLOWS:

For Patent Owner's Statement (Optional): TWO MONTHS from the mailing date of this communication (37 CFR 1.530 (b)). **EXTENSIONS OF TIME ARE GOVERNED BY 37 CFR 1.550(c).**

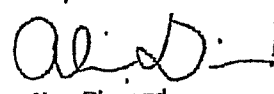
For Requester's Reply (optional): TWO MONTHS from the date of service of any timely filed Patent Owner's Statement (37 CFR 1.535). **NO EXTENSION OF THIS TIME PERIOD IS PERMITTED.** If Patent Owner does not file a timely statement under 37 CFR 1.530(b), then no reply by requester is permitted.

2. ☐ The request for *ex parte* reexamination is DENIED.

This decision is not appealable (35 U.S.C. 303(c)). Requester may seek review by petition to the Commissioner under 37 CFR 1.181 within ONE MONTH from the mailing date of this communication (37 CFR 1.515(c)). **EXTENSION OF TIME TO FILE SUCH A PETITION UNDER 37 CFR 1.181 ARE AVAILABLE ONLY BY PETITION TO SUSPEND OR WAIVE THE REGULATIONS UNDER 37 CFR 1.183.**

In due course, a refund under 37 CFR 1.26 (c) will be made to requester:

- a) ☐ by Treasury check or,
b) ☐ by credit to Deposit Account No. _____, or
c) ☐ by credit to a credit card account, unless otherwise notified (35 U.S.C. 303(c)).


Alan Diamond
Primary Examiner
Art Unit: 3991

cc:Requester (if third party requester)

U.S. Patent and Trademark Office
PTOL-471 (Rev. 08-05)

Office Action in *Ex Parte* Reexamination

Part of Paper No. 20070314

Application/Control Number: 90/008,360
Art Unit: 3991

Page 2

Discussion on Re examination Request

1. A Substantial new question of patentability affecting claims 1-24 of United States Patent Number 6,773,864 is raised by the request for *ex parte* reexamination.

Extension of Time

2. Extensions of time under 37 CFR 1.136(a) will not be permitted in these proceedings because the provisions of 37 CFR 1.136 apply only to "an applicant" and not to parties in a reexamination proceeding. Additionally, 35 U.S.C. 305 requires that *ex parte* reexamination proceedings "will be conducted with special dispatch" (37 CFR 1.550(a)). Extensions of time in *ex parte* reexamination proceedings are provided for in 37 CFR 1.550(c).

Substantial New Question of Patentability (SNQ)

3. The presence or absence of a "substantial new question of patentability" determines whether or not reexamination is ordered.

For a "substantial new question of patentability" to be present, it is only necessary that :

A) the prior art patents and/or printed publications raise a substantial new question of patentability regarding at least one claim, i.e., the teaching of the (prior art) patents and printed publications is such that a reasonable examiner would consider the teaching to be important in deciding whether or not the claim is patentable; and

Application/Control Number: 90/008,360
Art Unit: 3991

Page 3

B) the same question of patentability as to the claim has not been decided by the Office in a previous examination of the patent or in a final holding of invalidity by the Federal Courts in a decision on the merits involving the claim.

A SNQ may be based solely on old art where the old art is being presented/viewed in a new light, or in a different way, as compared with its use in the earlier concluded examination(s), in view of a material new argument or interpretation in the request. (MPEP 2242).

Request

4. Third Party Requestor relies on the following prior art references and printed publications:

- U.S. Patent No. 4,910,122 to Arnold et al. ("**Arnold**")
- Brewer, T., et al, "The Reduction of the Standing Wave Effect in Positive Photoresists," *Jour. Appl. Photogr. Eng.*, Vol. 7, no. 6, pp. 184-186 (Dec. 1981) ("**Brewer**")
- Crivello et al., *J. Polym. Sci.: Polym. Chem* 21 (1983) 97-109
- U.S. Patent No. 4,430,153 to Gleason ("**Gleason**")
- U.S. Patent No. 4,299,938 to Green ("**Green**")

Application/Control Number: 90/008,360

Art Unit: 3991

- U.S. Patent No. 4,863,827 to Jain, et al. ("Jain")
- U.S. Patent No. 3,884,702 to Koshimo, et al. ("Koshimo")
- Lamola, A., et al., "Chemically Amplified Resists", *Solid State Technology*, 53-60 (August 1991) ("Lamola")
- Y.-C. Lin, et al., "Some Aspects of Anti-Reflective Coating for Optical Lithography," *Advances in Resist Technology and Processing, Proc., SPIE* vol. 469, pp. 30-37 (1984) ("Lin")
- McKean, et al., "Characterization of a Novolac-Based Three-Component Deep-UV Resist," *Chem. Mater.* (1990) 2, 619-624 ("McKean")
- Nalamasu, et al., "Development of a Chemically Amplified Positive (CAMP) Resist Material for Single Layer Deep-UV Lithography," *Advances in Resist Technology and Processing VII*, SPIE 1262, 32-41 (1990) ("Nalamasu")
- U.S. Patent No. 4,935,320 to Rohde et al. ("Rohde")
- Silverstein, et al. "*Spectrometric Identification of Organic Compounds* John Wiley and Sons, 1991 pp. 309-311 ("Silverstein")
- Willson, C.G., "Organic Resist Materials - Theory and Chemistry", *Introduction to Microlithography*, American Chemical Society, 87-159 (1983) ("Willson")
- U.S. Patent No. 4,587,138 to Yau, et al. ("Yau")

Application/Control Number: 90/008,360
Art Unit: 3991

Page 5

As noted on page 4 of the request, none of the references except Arnold, Green, Jain and Lamola, were of record during prosecution of U.S. Patent 6,773,864.

5. The request indicates that Requestor considers claims 1 and 2 are unpatentable over any of Brewer, Gleason, Jain, Lin, Rohde, or Yau in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of any of Brewer, Gleason, Jain, Lin, Rohde, or Yau in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 1 and 2. Page 27, line 3 through page 29, line 22 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Gleason, Jain, Lin, Rohde, Yau, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 1 and 2 were patentable. Accordingly, any of Brewer, Gleason, Jain, Lin, Rohde, or Yau in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 1 and 2.

6. The request indicates that Requestor considers claim 3 is unpatentable over any of Brewer, Gleason, Lin, Rohde, or Yau in view of any of Lamola or McKean or Nalamasu or Willson.

Application/Control Number: 90/008,360
Art Unit: 3991

Page 6

It is agreed that consideration of any of Brewer, Gleason, Lin, Rohde, or Yau in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claim 3. Page 29, line 23 through page 30, line 11 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Gleason, Lin, Rohde, Yau, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claim 3 was patentable. Accordingly, any of Brewer, Gleason, Lin, Rohde, or Yau in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claim 3.

7. The request indicates that Requestor considers claim 4, 6, 7, 15 and 17 are unpatentable over either Jain or Rohde in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of either Jain or Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 4, 6, 7, 15 and 17. Page 30, lines 12 through 26, page 31, line 10 through page 32, line 18, page 39, lines 1 through 14, and page 40, lines 1 through 18 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Jain, Rohde, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There

Application/Control Number: 90/008,360
Art Unit: 3991

Page 7

is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 4, 6, 7, 15 and 17 were patentable.

Accordingly, either Jain or Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 4, 6, 7, 15 and 17.

8. The request indicates that Requestor considers claims 5, 8, 16 and 19 are unpatentable over Rohde in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 5, 8, 16 and 19. Page 30, line 27 through page 31, line 9, page 32, line 23 through page 33, line 3, page 39, lines 15 through 28, and page 41, lines 2 through 13 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Rohde, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 5, 8, 16 and 19 were patentable. Accordingly, Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 5, 8, 16 and 19.

Application/Control Number: 90/008,360
Art Unit: 3991

Page 8

9. The request indicates that Requestor considers claims 8, 18 and 19 are unpatentable over Jain in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of Jain in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 8, 18 and 19. Page 33, lines 3 through 10, page 40, line 19 through page 41, line 1, and page 41, lines 14 through 21 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Jain, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 8, 18 and 19 were patentable. Accordingly, Jain in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 8, 18 and 19.

10. The request indicates that Requestor considers claims 8 and 19 are unpatentable over any of Gleason, Lin or Brewer in view of Koshimo and any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of any of Gleason, Lin or Brewer in view of Koshimo and any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 8 and 19. Page 33, lines 11 through 19, and page 41, line 22 through page 42, line 2 of the request for reexamination are hereby

Application/Control Number: 90/008,360

Page 9

Art Unit: 3991

incorporated by reference for their explanation of the teaching provided in Gleason, Lin, Brewer, Koshimo, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 8 and 19 were patentable. Accordingly, any of Gleason, Lin or Brewer in view of Koshimo and any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 8 and 19.

11. The request indicates that Requestor considers claim 8 is unpatentable over any of Gleason, Lin, Brewer or Jain¹ in view of any of Lamola or McKean or Nalamasu or Willson and in further view of Jain or Arnold in view of Silverstein.

It is agreed that consideration of any of Gleason, Lin, Brewer or Jain in view of any of Lamola or McKean or Nalamasu or Willson and in further view of Jain or Arnold in view of Silverstein raises a substantial new question of patentability as to claim 8.

Page 33, line 20 through page 34, line 5 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Gleason, Lin, Brewer, Jain, Lamola, McKean, Nalamasu, Willson, Arnold and Silverstein that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claim 8 was patentable. Accordingly, any of

¹ Page 33, lines 20-23, recite "Claims 8/1, 8/2, 8/4, 8/6 And 8/7 Are Obvious Over Any Of Gleason, Lin, Brewer In View Of Any Of Lamola Or McKean Or Nalamasu Or Willson And In Further View Of Jain Or Arnold In View Of Silverstein." The term "Brewer In View Of" in this recitation should clearly have been "Brewer Or Jain In View Of" based on the teachings at page 33, line 24 through page 25, line 5 of the Request.

Application/Control Number: 90/008,360
Art Unit: 3991

Page 10

Gleason, Lin, Brewer or Jain in view of any of Lamola or McKean or Nalamasu or Willson and in further view of Jain or Arnold in view of Silverstein raises a substantial new question of patentability as to claim 8.

12. The request indicates that Requestor considers claim 9 is unpatentable over any of Brewer, Gleason, Jain or Rohde in view of Crivello or Green '938, each in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of any of Brewer, Gleason, Jain or Rohde in view of Crivello or Green '938, each in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claim 9. Page 34, line 18 through page 35, line 14 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Gleason, Jain, Rohde, Crivello, Green '938, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claim 9 was patentable. Accordingly, any of Brewer, Gleason, Jain or Rohde in view of Crivello or Green '938, each in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claim 9.

Application/Control Number: 90/008,360

Page 11

Art Unit: 3991

13. The request indicates that Requestor considers claims 10 and 23 unpatentable over any of Brewer, Gleason or Lin in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of any of Brewer, Gleason or Lin in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 10 and 23. Page 35, line 15 through page 36, line 9, and page 45, lines 12 through 30 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Gleason, Lin, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 10 and 23 were patentable. Accordingly, any of Brewer, Gleason or Lin in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 10 and 23.

14. The request indicates that Requestor considers claims 11-13, 20 and 24 are unpatentable over any of Brewer, Gleason, Jain, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of any of Brewer, Gleason, Jain, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 11-13, 20 and 24. Page 36, line 10 through page 38, line 12, page 42, line 21 through page 43, line 13, and page 46, lines 1 through 19

Application/Control Number: 90/008,360
Art Unit: 3991

Page 12

of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Gleason, Jain, Lin, Rohde, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 11-13, 20 and 24 were patentable. Accordingly, any of Brewer, Gleason, Jain, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 11-13, 20 and 24.

15. The request indicates that Requestor considers claim 14 is unpatentable over any of Brewer, Gleason, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson.

It is agreed that consideration of any of Brewer, Gleason, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claim 14. Page 38, lines 13 through 30 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Gleason, Lin, Rohde, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claim 14 was patentable. Accordingly, any of Brewer, Gleason, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claim 14.

Application/Control Number: 90/008,360
Art Unit: 3991

Page 13

16. The request indicates that Requestor considers claim 19 is unpatentable over any of Gleason, Lin, Brewer or Jain² in view of any of Lamola or McKean or Nalamasu or Willson and in further view of Jain and Arnold.

It is agreed that consideration of any of Gleason, Lin, Brewer or Jain in view of any of Lamola or McKean or Nalamasu or Willson and in further view of Jain and Arnold raises a substantial new question of patentability as to claim 19. Page 42, lines 3 through 11 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Gleason, Lin, Brewer, Jain, Lamola, McKean, Nalamasu, Willson, and Arnold that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claim 19 was patentable. Accordingly, any of Gleason, Lin, Brewer or Jain in view of any of Lamola or McKean or Nalamasu or Willson and in further view of Jain and Arnold raises a substantial new question of patentability as to claim 19.

17. The request indicates that Requestor considers claims 21 and 22 are unpatentable over Gleason in view of any of Lamola or McKean or Nalamasu or Willson.

² Page 42, lines 3-6, recite "Claims 19/12, 19/13, 19/15, 19/16 And 19/17 Are Obvious Over Any Of Gleason, Lin, Brewer Or In View Of Any Of Lamola Or McKean Or Nalamasu Or Wilson And In Further View Of Jain Or Arnold." The term "Brewer Or In View Of" in this recitation should clearly have been "Brewer Or Jain In View Of" based on the teachings at lines 7-11 on page 42 of the Request.

Application/Control Number: 90/008,360
Art Unit: 3991

Page 14

It is agreed that consideration of Gleason in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 21 and 22. Page 43, lines 15 through 23, and page 44, lines 15 through 21 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Gleason, Lamola, McKean, Nalamasu and Willson that was not present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 21 and 22 were patentable. Accordingly, Gleason in view of any of Lamola or McKean or Nalamasu or Willson raises a substantial new question of patentability as to claims 21 and 22.

18. The request indicates that Requestor considers claims 21 and 22 are unpatentable over Brewer, Jain, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson or in view of Lamola or McKean or Nalamasu and in further view of Gleason or Arnold.

It is agreed that consideration of Brewer, Jain, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson or in view of Lamola or McKean or Nalamasu and in further view of Gleason or Arnold raises a substantial new question of patentability as to claims 21 and 22. Page 43, line 24 through page 44, line 5, and page 44, line 21 through page 45, line 6 of the request for reexamination are hereby incorporated by reference for their explanation of the teaching provided in Brewer, Jain, Lin, Rohde, Lamola, McKean, Nalamasu, Willson, Gleason and Arnold that was not

Application/Control Number: 90/008,360
Art Unit: 3991

Page 15

present in the prosecution of the application which became the 6,773,864 patent. There is a substantial likelihood that a reasonable examiner would consider this teaching important in deciding whether or not claims 21 and 22 were patentable. Accordingly, Brewer, Jain, Lin or Rohde in view of any of Lamola or McKean or Nalamasu or Willson or in view of Lamola or McKean or Nalamasu and in further view of Gleason or Arnold raises a substantial new question of patentability as to claims 21 and 22.

Use of Old Art

19. The above SNQs with respect to Jain in view of Lamola (claims 1, 2, 4, 6, 7, 8, 11-13, 15, 17-20 and 24), Jain in view of Lamola and further in view of Jain or Arnold (claim 19), and Jain in view of Lamola or in view of Lamola and in further view of Arnold (claims 21 and 22) are based solely on patents and/or printed publications already cited/considered in an earlier concluded examination of the patent being reexamined. On November 2, 2002, Public Law 107-273 was enacted. Title III, Subtitle A, Section 13105, part (a) of the Act revised the reexamination statute by adding the following new last sentence to 35 U.S.C. 303(a) and 312(a):

"The existence of a substantial new question of patentability is not precluded by the fact that a patent or printed publication was previously cited by or to the Office or considered by the Office."

For any reexamination ordered on or after November 2, 2002, the effective date of the statutory revision, reliance on previously cited/considered art, i.e., "old art," does not necessarily preclude the existence of a substantial new question of patentability

Application/Control Number: 90/008,360
Art Unit: 3991

Page 16

(SNQ) that is based exclusively on that old art. Rather, determinations on whether a SNQ exists in such an instance shall be based upon a fact-specific inquiry done on a case-by-case basis.

In the present instance, there exists a SNQ based on the Jain, Lamola and Arnold references. A discussion of the specifics now follows: None of Jain, Lamola or Arnold was relied upon to reject any claims during prosecution of Application Serial No. 10/335,476, which became U.S. Patent 6,773,864. Application Serial No. 10/335,476 was allowed after Patent Owner, in the amendment of 03/01/2004, amended the independent claims in the case to recite that the coating layer of photoresist is a "chemically-amplified positive" photoresist, and argued this limitation as a distinguishing feature. As pointed out on pages 27-28 of the request, Jain teaches the instant substrate and the instant antihalation layer comprising a silicon-containing material under a photoresist. Jain does not teach a chemically-amplified positive photoresist. However, it is Lamola that teaches a chemically amplified photoresist (request, pages 27-28), which is said to be useful in deep-UV applications to improve resolution of narrow line widths.

With respect to the anthracene material in dependent claim 19, Jain teaches that its photosensitizer (dye) can be 1,2-anthraquinone-2-diazide-4-sulfonyl (col. 10, lines 26-27). Also with respect to claim 19, Arnold teaches that dyes which are used are those that absorb in the wavelength region of the imaging source (col. 4, lines 13-27). These teachings are important because it is well known that anthracene is highly absorbent in said deep-UV (request, page 19).

Application/Control Number: 90/008,360
Art Unit: 3991

Page 17

Dependent claims 21 and 22 respectively recite that areas bared of photoresist upon treatment are etched, and that areas bared of photoresist upon treatment with the developer are exposed to a plasma gas. Jain discloses exposure of the photoresist composition to activating energy, which exposure is followed by treatment with a developer to produce a relief image (request, pages 43 and 44). It is Arnold that teaches etching areas bared of photoresist (request, page 44) and etching of areas bared of photoresist using a plasma gas (request, page 45).

Duty to Disclose

20. The patent owner is reminded of the continuing responsibility under 37 CFR 1.565(a) to apprise the Office of any litigation activity, or other prior or concurrent proceeding, involving Patent No. 6,773,864 throughout the course of this reexamination proceeding. The third party requester is also reminded of the ability to similarly apprise the Office of any such activity or proceeding throughout the course of this reexamination proceeding. See MPEP §§ 2207, 2282 and 2286.

Correspondence

21. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Alan Diamond whose telephone number is (571) 272-1338. The examiner can normally be reached on Monday through Friday from 5:30 a.m. to 2:00 p.m.

If attempts to reach the examiner by telephone are unsuccessful; the examiner's supervisor, Deborah Jones can be reached on (571) 272-1535.

Application/Control Number: 90/008,360

Page 18

Art Unit: 3991

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

Please mail any communications to:
Attn: Mail Stop "Ex Parte Reexam"
Central Reexamination Unit
Commissioner for Patents
P. O. Box 1450
Alexandria VA 22313-1450

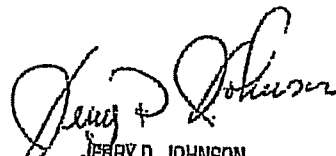
Please FAX any communications to:
(571) 273-9900
Central Reexamination Unit

Please hand-deliver any communications to:
Customer Service Window
Attn: Central Reexamination Unit
Randolph Building, Lobby Level
401 Dulany Street
Alexandria, VA 22314

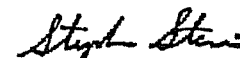
Signed:



Alan Diamond
Primary Examiner
Central Reexamination Unit
Art Unit 3991
(571) 272-1338



JERRY D. JOHNSON
CRU EXAMINER-AU 3991



STEPHEN J. STEIN
CRU EXAMINER - AU 3991

EXHIBIT N - REDACTED

EXHIBIT O

Daniel Mulveny

From: ded_nefreply@ded.uscourts.gov
Sent: Tuesday, July 10, 2007 11:03 AM
To: ded_ecf@ded.uscourts.gov
Subject: Activity in Case 1:06-cv-00297-GMS Rohm and Haas Electronic Materials LLC v. Honeywell Electronic Materials Inc. et al Oral Order

This is an automatic e-mail message generated by the CM/ECF system. Please DO NOT RESPOND to this e-mail because the mail box is unattended.
*****NOTE TO PUBLIC ACCESS USERS*** You may view the filed documents once without charge. To avoid later charges, download a copy of each document during this first viewing.**

U.S. District Court

District of Delaware

Notice of Electronic Filing

The following transaction was entered on 7/10/2007 at 11:02 AM EDT and filed on 7/10/2007
Case Name: Rohm and Haas Electronic Materials LLC v. Honeywell Electronic Materials Inc.
et al

Case Number: 1:06-cv-297

Filer:

Document
Number: No document attached

Docket Text:

ORAL ORDER that the court WILL NOT entertain any additional stipulations to extend time regarding submission of the joint claim chart. Ordered by Judge Gregory M. Sleet on 7/10/2007. (asw)

1:06-cv-297 Notice has been electronically mailed to:

Rudolf E. Hutz rhutz@cblh.com, dkt@cblh.com
Robert Scott Saunders rsaunder@skadden.com, ckrebs@skadden.com, mstow@skadden.com
Daniel Christopher Mulveny dmulveny@cblh.com
James Darlington Taylor, Jr james.taylor@bipc.com
Constance S. Huttner constance.huttner@bipc.com

1:06-cv-297 Notice has been delivered by other means to:

EXHIBIT P

Daniel Mulveny

From: Daniel Mulveny
Sent: Tuesday, July 10, 2007 11:10 AM
To: 'Jacobson, Scott'; Huttner, Constance
Cc: Darryl Frickey; Votava, Shannon M
Subject: RE: update – Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Thanks for the update Scott.

Everyone:

As you may know, we just got an oral order from the Court that no more extensions of time will be granted, so we need to wrap this up as soon as possible.

Best regards,
Dan

REDACTED

Dan

Daniel C. Mulveny
Connolly Bove Lodge & Hutz LLP
The Nemours Building
1007 North Orange Street
Wilmington, DE 19801
(302) 884-6593 direct telephone
(302) 661-2331 telefax

<http://www.cblhlaw.com/>

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

EXHIBIT Q

Daniel Mulveny

From: Jacobson, Scott [Scott.Jacobson@Honeywell.com]
Sent: Wednesday, July 11, 2007 5:51 PM
To: Daniel Mulveny
Cc: Jacobson, Scott
Subject: RE: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Same. I have sent another message and voice mail to Connie, but I have not been able to reach her. At this point it looks like she will be getting this to me after my close of business, so assuming she gets it to me in the morning, I will be able to turn it around tomorrow afternoon (I am out of the office tomorrow morning).

I appreciate your patience and feel as strongly as you do to get this done.

Thanks, Scott

Scott D. Jacobson
Intellectual Property Counsel, Specialty Materials Honeywell International Inc.
101 Columbia Road
Morristown, NJ 07962
Building Nichols-4
973-455-2013 (direct)
973-455-6199 (fax)

-----Original Message-----

From: Daniel Mulveny [mailto:DMulveny@cb1h.com]
Sent: Wednesday, July 11, 2007 5:31 PM
To: Huttner, Constance; Jacobson, Scott
Subject: RE: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Connie and Scott:

What is the status?

Dan

-----Original Message-----

From: Huttner, Constance [mailto:constance.huttner@bipc.com]
Sent: Wednesday, July 11, 2007 10:43 AM
To: Daniel Mulveny; Scott.Jacobson@Honeywell.com
Subject: Re: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Dan, Scott is waiting on me and I have been out of town. I will get him my comments this am and I am sure he will get you the collective comments later today. I am sorry for being the source of the delay.

----- Original Message -----

From: Daniel Mulveny <DMulveny@cb1h.com>
To: Jacobson, Scott <Scott.Jacobson@Honeywell.com>; Huttner, Constance
Cc: Darryl Frickey <DFrickey@rohmmaas.com>; Votava, Shannon M <Shannon.Votava@Honeywell.com>
Sent: Wed Jul 11 10:30:23 2007
Subject: RE: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Scott:

Please advise when you will be send us your comments.

Best regards,
Dan

REDACTED

) REDACTED

1

REDACTED

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.
If you are not the intended recipient, please advise the sender immediately.
Unauthorized use or distribution is prohibited and may be unlawful.

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

EXHIBIT R

Daniel Mulveny

From: Huttner, Constance [constance.huttner@bipc.com]
Sent: Wednesday, July 11, 2007 8:57 PM
To: Daniel Mulveny
Cc: Jacobson, Scott
Subject: RE: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

yes, the problem is me. i have been unexpectedly and unhappily under water all day long, and just was not able to get through it until now. mostly, i am just trying to clean up stray typos, inconsistent definitions and the like. you will have it tomorrow. please accept my apologies for the delay.

Constance S. Huttner
Chair Intellectual Property Litigation Section
Buchanan, Ingersoll & Rooney, P.C.
One Chase Manhattan Plaza
New York, New York 10005-1417
(212) 440-4426
constance.huttner@bipc.com

From: Daniel Mulveny [mailto:DMulveny@cblh.com]
Sent: Wednesday, July 11, 2007 7:52 PM
To: Huttner, Constance
Subject: Re: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Connie:

Is there something causing the holdup?

Dan

-----Original Message-----

From: Huttner, Constance [mailto:constance.huttner@bipc.com]
Sent: Wednesday, July 11, 2007 07:01 PM Eastern Standard Time
To: Daniel Mulveny
Subject: Re: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

It is still my fault

----- Original Message -----

From: Daniel Mulveny <DMulveny@cblh.com>
To: Huttner, Constance; Scott.Jacobson@Honeywell.com <Scott.Jacobson@Honeywell.com>
Sent: Wed Jul 11 17:30:34 2007
Subject: RE: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Connie and Scott:

What is the status?

Dan

-----Original Message-----

From: Huttner, Constance [mailto:constance.huttner@bipc.com]
Sent: Wednesday, July 11, 2007 10:43 AM
To: Daniel Mulveny; Scott.Jacobson@Honeywell.com

Subject: Re: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Dan, Scott is waiting on me and I have been out of town. I will get him my comments this am and I am sure he will get you the collective comments later today. I am sorry for being the source of the delay.

----- Original Message -----

From: Daniel Mulveny <DMulveny@cblh.com>
To: Jacobson, Scott <Scott.Jacobson@Honeywell.com>; Huttner, Constance
Cc: Darryl Frickey <DFrickey@rohmmaas.com>; Votava, Shannon M <Shannon.Votava@Honeywell.com>
Sent: Wed Jul 11 10:30:23 2007
Subject: RE: update -- Final RHEM-Honeywell Settlement and Patent License Agreement.DOC

Scott:

Please advise when you will be send us your comments.

Best regards,
Dan

REDACTED

REDACTED

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.
If you are not the intended recipient, please advise the sender immediately.
Unauthorized use or distribution is prohibited and may be unlawful.

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.
If you are not the intended recipient, please advise the sender immediately.
Unauthorized use or distribution is prohibited and may be unlawful.

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.
If you are not the intended recipient, please advise the sender immediately.

Unauthorized use or distribution is prohibited and may be unlawful.

EXHIBIT S - REDACTED

EXHIBIT T

Daniel Mulveny

From: Jacobson, Scott [Scott.Jacobson@Honeywell.com]
Sent: Friday, July 13, 2007 5:46 PM
To: Daniel Mulveny; dfickey@rohmhaas.com
Cc: Huttner, Constance; Jacobson, Scott; Votava, Shannon M
Subject: RE: Settlement Agmt edits

Dan, I am leaving the office now, so I will look for the next version from you next week. Please note that I will be out of the office and unavailable from Thursday of next week until July 30. As soon as I get the next version from you, we should discuss as soon as possible and try to finalize before cob Wednesday. Thanks, Scott

Scott D. Jacobson
Intellectual Property Counsel, Specialty Materials
Honeywell International Inc.
101 Columbia Road
Morristown, NJ 07962
Building Nichols-4
973-455-2013 (direct)
973-455-6199 (fax)

REDACTED

EXHIBIT U - REDACTED

EXHIBIT V - REDACTED

EXHIBIT W

Daniel Mulveny

From: Daniel Mulveny
Sent: Monday, July 16, 2007 6:32 PM
To: 'Jacobson, Scott'; dfickey@rohmhaas.com
Cc: Huttner, Constance; Votava, Shannon M
Subject: RE: Settlement Agmt edits

Scott:

We've been trying to contact you and Connie regarding feedback on the latest version I sent this morning. I spoke to Connie on the phone earlier today my understanding is that we're in agreement as to the terms. If that is the case, please let us know.

I would like to call the Court tonite to leave a message that an agreement has been reached and that the parties will be working to execute the agreement tomorrow. Because were to otherwise be filing a joint claim chart, as a courtesy to the Court, we should notify the Court what is going on regarding the settlement.

I have to leave the office now, but if you need to reach me, please either email or call my cell phone: (302) 530-4830.

Best regards,
Dan

REDACTED

REDACTED

EXHIBIT X

RE:

Daniel Mulveny

From: Taylor, Jr., James D. [james.taylor@bipc.com]
Sent: Wednesday, July 18, 2007 10:13 AM
To: Daniel Mulveny
Subject: RE:

let's call now - what's your direct line?

James D. Taylor, Jr., Esquire
Buchanan Ingersoll & Rooney PC
1000 West Street, Suite 1410
P.O. Box 1397
Wilmington, Delaware 19899-1397
Telephone: (302) 552-4237
Facsimile: (302) 552-4295
E-mail: james.taylor@bipc.com

This message is intended for the exclusive use of the named addressee and may contain information that is privileged or confidential or otherwise legally exempt from disclosure. If you have received this message in error or are not the named addressee or his or her authorized agent, please notify me immediately by e-mail, discard any paper copies and delete all electronic files of this message.

From: Daniel Mulveny [mailto:DMulveny@cblh.com]
Sent: Wednesday, July 18, 2007 9:31 AM
To: Taylor, Jr., James D.
Subject: RE:

I have not heard from Honeywell when to expect the signed agreement from them. What is your availability today to call the court?

-----Original Message-----

From: Taylor, Jr., James D. [mailto:james.taylor@bipc.com]
Sent: Wednesday, July 18, 2007 09:28 AM Eastern Standard Time
To: Daniel Mulveny
Subject: RE:

Dan, I received your voicemail and agree that a call is in order unless we can file a stipulation promptly. Have you been given an eta for the signed agreement from Honeywell?

James D. Taylor, Jr., Esquire
Buchanan Ingersoll & Rooney PC
1000 West Street, Suite 1410
P.O. Box 1397
Wilmington, Delaware 19899-1397
Telephone: (302) 552-4237
Facsimile: (302) 552-4295
E-mail: james.taylor@bipc.com <<mailto:james.taylor@bipc.com>>

This message is intended for the exclusive use of the named addressee and may contain information that is privileged or confidential or otherwise legally exempt from disclosure. If you have received this

RE:

message in error or are not the named addressee or his or her authorized agent, please notify me immediately by e-mail, discard any paper copies and delete all electronic files of this message.

From: Daniel Mulveny [<mailto:DMulveny@cblh.com>]
Sent: Wednesday, July 18, 2007 8:15 AM
To: Huttner, Constance
Cc: Jacobson, Scott; Votava, Shannon M; Taylor, Jr., James D.
Subject: RE:

Connie:

I will work it out with Jim to contact the Court regarding settlement and filing a Stipulation of Dismissal once we receive the signed agreement from Scott.

Dan

From: Huttner, Constance [<mailto:constance.huttner@bipc.com>]
Sent: Wednesday, July 18, 2007 6:09 AM
To: Daniel Mulveny
Cc: Jacobson, Scott; Votava, Shannon M; Taylor, Jr., James D.
Subject:

Dan, I will be available for a few hours this morning, and for limited periods throughout the day today. Perhaps you and Jim should call Judge Sleet this morning and tell him we have reached a settlement. Once you receive the signed agreement from Scott, we can make arrangements to file the Stipulation of Dismissal with the Court. You can reach me this morning at 212 46 2080. Thanks.

Constance S. Huttner
Chair Intellectual Property Litigation Section
Buchanan, Ingersoll & Rooney, P.C.
One Chase Manhattan Plaza
New York, New York 10005-1417
(212) 440-4426
constance.huttner@bipc.com

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.

RE:

If you are not the intended recipient, please advise the sender immediately.

Unauthorized use or distribution is prohibited and may be unlawful.

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.
If you are not the intended recipient, please advise the sender immediately.
Unauthorized use or distribution is prohibited and may be unlawful.

This e-mail and any attachment is intended only for use by the addressee(s) and may contain privileged and confidential information. Please email or call 302-658-9141 to advise the sender of a transmission error and delete the message and any attachments and any printouts. Any tax advice contained in this communication is not intended and cannot be used to avoid penalties under the Internal Revenue Code or to promote, market or recommend to another party any transaction or matter addressed herein.

TAX ADVICE DISCLAIMER: Any federal tax advice contained in this communication (including attachments) was not intended or written to be used, and it cannot be used, by you for the purpose of (1) avoiding any penalty that may be imposed by the Internal Revenue Service or (2) promoting, marketing or recommending to another party any transaction or matter addressed herein. If you would like such advice, please contact us.

Above email is for intended recipient only and may be confidential and protected by attorney/client privilege.

If you are not the intended recipient, please advise the sender immediately.

Unauthorized use or distribution is prohibited and may be unlawful.

EXHIBIT Y

**IN THE UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF DELAWARE**

ROHM AND HAAS ELECTRONIC
MATERIALS LLC

Plaintiff,

Y.

HONEYWELL ELECTRONIC
MATERIALS INC.
HONEYWELL SPECIALTY
MATERIALS, LLC
and
HONEYWELL INTERNATIONAL INC.,

Defendants.

[illegible]

Civil Action No. 06-297-GMS

**ANSWER OF HONEYWELL ELECTRONIC MATERIALS INC., HONEYWELL
SPECIALTY MATERIALS, LLC AND HONEYWELL INTERNATIONAL INC.,
AND COUNTERCLAIMS OF HONEYWELL INTERNATIONAL INC.**

Defendants Honeywell Electronic Materials Inc., Honeywell Specialty Materials, LLC and Honeywell International Inc. (respectively "HEM", "HSM" and "Honeywell International"; collectively, "Honeywell") for their Answer to the Complaint of Plaintiff Rohm and Haas Electronic Materials LLC ("Plaintiff" or "Rohm and Haas") in this action, and Honeywell International, for its Counterclaims, hereby state as follows:

PARTIES

1. Honeywell is without sufficient information to form a belief as to the truth of the allegations in paragraph 1 of Plaintiff's Complaint and, therefore, denies the allegations in that paragraph.

17. Honeywell denies all allegations in Plaintiff's Complaint that are not otherwise specifically admitted in this Answer and Counterclaims.

AFFIRMATIVE DEFENSES

18. The Complaint fails to state a claim upon which relief may be granted.

19. As to HEM and HSM, the Complaint fails to state a claim upon which relief may be granted because these entities lack a factual and legal nexus to the alleged infringing activity and, therefore, are not proper parties to the action.

20. Honeywell has not infringed and is not infringing, either literally or under the doctrine of equivalents, any claim of either the '128 or '864 patents.

21. Honeywell has not contributorily infringed and is not contributorily infringing, and Honeywell International has not actively induced infringement and is not actively inducing others to infringe any claim of either the '128 or '864 patents.

22. The '128 and '864 patents, which the Complaint alleges to have been infringed by Honeywell, are invalid and void for failing to meet the requirements of Title 35 of the U.S. Code, including, but not limited to, Sections 102, 103 and 112 thereof.

23. Plaintiff's infringement action against Honeywell is barred under the equitable doctrines of laches and estoppel.

24. The '128 and '864 patents are unenforceable under the equitable doctrine of prosecution laches.

COUNTERCLAIMS

For its Counterclaims, Honeywell International alleges as follows:

25. These Counterclaims arise under the patent laws of the United States, 35 U.S.C. § 1, *et seq.*, including 35 U.S.C. § 271, and the Federal Declaratory Judgment Act, 28

First Counterclaim

(Declaratory Judgment – Non-infringement)

33. Honeywell International realleges and incorporates herein by reference each of paragraphs 1-32 above.

34. Honeywell International has not infringed and is not infringing any claim, either literally or under the doctrine of equivalents, of the '128 or '864 patents. Honeywell International has not contributorily infringed and is not contributorily infringing, and has not actively induced infringement and is not actively inducing others to infringe any claim of either the '128 or '864 patents.

35. Honeywell International seeks a judicial declaration that it does not infringe and has not infringed, directly or indirectly, literally or by equivalents, any claim of either the '128 or '864 patents.

Second Counterclaim

(Declaratory Judgment – Invalidity)

36. Honeywell International realleges and incorporates herein by reference each of paragraphs 1-35 above.

37. The '128 and '864 patents are invalid and void for failure to comply with the conditions of patentability specified in one or more sections of Title 35 of the United States Code, including, but not limited to, Sections 102, 103 and 112 thereof.

38. Honeywell International seeks a judicial declaration that the '128 and '864 patents are invalid.

EXHIBIT Z - REDACTED

EXHIBIT AA

DATED: December 12, 2006

preliminary injunctive relief"); *Gioello Enters. Ltd. v. Mattel, Inc.*, No. C.A. 99-375-GMS, 2001 WL 125340, at *2 (D. Del. Jan. 29, 2001) (finding that where the plaintiff was not selling or licensing goods or services related to the patent at issue, "money damages [were] an adequate remedy for any delay in redress").

Finally, since the Plaintiff brought suit against Honeywell seeking protection under the patent statutes, it "can hardly be heard now to complain of the rights afforded others by that same statutory framework." *Pegasus Dev. Corp. v. DirecTV, Inc.*, No. 00-1020, 2003 U.S. Dist. LEXIS 8052, at *8 (D. Del. May 14, 2003).

2. A Stay Will Simplify The Issues In Question And May Dispose Of The Case Entirely

A stay is also warranted because it will simplify the issues to be decided in this case. The overwhelming likelihood is that the Patent claims will be materially changed or voided after reexamination is completed. According to the PTO's Performance and Accountability Report, in 2006, the PTO granted 425 requests for *ex parte* reexamination and denied only 28 requests (a grant rate of approximately 94%). *See Exhibit E.* Similarly in 2005, 511 requests were granted while only 24 were denied (a grant rate of approximately 96%). *See id.* Indeed, from 1981 through September 30, 2006, the PTO granted 91% all reexamination requests submitted. *See Exhibit D.* Moreover, of the *ex parte* reexaminations conducted over this twenty-five year period, 74% of the reexaminations resulted in the cancellation or modification of at least some of the claims at issue. *See id.*

Since the claims of the Patents are likely to change as a result of reexamination (if they survive at all), there can be no real question that a stay will avoid the likelihood of enormous waste of the parties' and Court's resources. If the claims at

issue in this case are invalidated, as Honeywell expects they will be, the related allegations of infringement will be moot. If the claims are modified, the related allegations of infringement may also be moot, and at a minimum, the issues surrounding claim construction, validity, infringement, and damages will likely change. A stay will therefore benefit the Court and the parties by avoiding time, effort, and expense wasted on issues that are likely to be irrelevant, or at least different, following reexamination. *See Abbott Diabetes Care, Inc.*, 2006 U.S. Dist. LEXIS 57469, at *19 (“the court, as well as the parties, would benefit from a narrowing of the variety of complex issues relating to the numerous claims at issue, which, if clearly defined [during reexamination], would streamline the discovery process and the remainder of the litigation”); *Alloc, Inc. v. Unilin Decor N.V.*, No. 03-253-GMS, 2003 U.S. Dist. LEXIS 11917, at *7 (D. Del. July 11, 2003) (finding a stay pending reexamination beneficial for a variety of reasons, including that “many discovery issues relating to prior art may be alleviated” and that “the issues, defenses, and evidence will be more easily limited in pre-trial conferences following a reexamination”). A stay may also benefit third parties, including Intel Corporation, Honeywell’s primary customer for the accused products, by preventing such parties from having to provide potentially unnecessary discovery.

Additionally, if litigation proceeds in parallel with the reexamination proceedings, there is a risk that the PTO and the Court will arrive at different judgments concerning the validity and appropriate scope of the Patent claims which will confuse, rather than simplify, the issues to be decided. *See Gioello Enters. Ltd.*, 2001 WL 125340, at *1 (“Not staying the proceedings runs the risk of inconsistent adjudications or issuance of advisory opinions.”).

EXHIBIT BB - REDACTED

EXHIBIT CC

JUL-19-2007 THU 05:20 PM E A P & D

FAX NO. 6174394170

P. 04.

Docket No.: 40678-5C
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application:
Thackeray et al.

Application No.: 90/008,359

Confirmation No.: 8757

Filed: December 4, 2006

Art Unit: 3991

For: ANTIHALATION COMPOSITIONS

Examiner: S. Stein

SUPPLEMENTAL AMENDMENT

MS *Ex Parte* Reexam
ATTN: Central Reexamination Unit
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

INTRODUCTORY COMMENTS

On June 11, 2007, Applicants file a response to the Office Action mailed from the United States Patent and Trademark Office on April 9, 2007 in the above-identified application.

By the present Supplemental Amendment, Applicants supplement that response of June 11.

Amendments to the Claims are reflected in the listing of claims which begins on page 2 of this paper.

Remarks begin on page 6 of this paper.

JUL-19-2007 THU 05:21 PM E A P & D

FAX NO. 6174394170

P. 05

Application No. 90/008,359

2

Docket No.: 40678-5C

Amendments to the Claims

(currently amended) Claim 1. A coated substrate comprising:
a substrate having thereon:
a coating layer of an antireflective composition, the antireflective composition comprising a crosslinker and an anthracene material; and
a coating layer of a positive-acting photoresist composition over the antireflective composition coating layer.

(original) Claim 2. The substrate of claim 1 wherein the antireflective composition coating layer is crosslinked.

(original) Claim 3. The substrate of claim 1 wherein the antireflective composition comprises a thermal acid generator.

(original) Claim 4. The substrate of claim 1 wherein the substrate is a microelectronic wafer.

(currently amended) Claim 5. A method for forming a relief image on a substrate comprising:
applying on the substrate a layer of an antihalation composition comprising an anthracene material;
applying over the antihalation composition coating layer a positive-acting photoresist composition.

(original) Claim 6. The method of claim 5 wherein the antihalation composition coating layer is crosslinked prior to applying the photoresist composition.

(original) Claim 7. The method of claim 6 wherein the photoresist composition is imaged with activating radiation and the imaged photoresist composition is treated

JUL-19-2007 THU 05:21 PM E A P & D

FAX NO. 6174394170

P. 06

Application No. 90/008,359

3

Docket No.: 40678-5C

with a developer to provide a photoresist relief image.

(original) Claim 8. The method of claim 7 wherein areas bared of photoresist upon treatment with the developer are etched.

(original) Claim 9. The method of claim 7 wherein areas bared of photoresist upon treatment with the developer are exposed to a plasma gas.

(original) Claim 10. The method of claim 9 wherein the plasma gas penetrates the antihalation composition coating layer.

(original) Claim 11. The method of claim 5 wherein the antihalation composition comprises a thermal acid generator.

(original) Claim 12. The method of claim 5 wherein the substrate is a microelectronic wafer.

(original) Claim 13. The method of claim 5 wherein the photoresist composition is imaged with activating radiation and the imaged photoresist composition is treated with a developer to provide a photoresist relief image.

(original) Claim 14. The method of claim 13 wherein areas bared of photoresist upon treatment with the developer are etched.

(original) Claim 15. The method of claim 13 wherein areas bared of photoresist upon treatment with the developer are exposed to a plasma gas.

(original) Claim 16. The method of claim 15 wherein the plasma gas penetrates the antihalation composition coating layer.

(new) Claim 17. The method of claim 6 wherein the photoresist composition is imaged with radiation having a wavelength of 100 nm to 300 nm.

JUL-19-2007 THU 05:21 PM E A P & D

FAX NO. 6174394170

P. 07

Application No. 90/008,359

4

Docket No.: 40678-5C

(new) Claim 18. The method of claim 6 wherein the photoresist composition is imaged with radiation having a wavelength of 248 nm.

(new) Claim 19. The method of claim 7 wherein the photoresist layer is imaged with radiation having a wavelength of 100 to 300 nm.

(new) Claim 20. The method of claim 7 wherein the photoresist layer is imaged with radiation having a wavelength of 248 nm.

(new) Claim 21. The method of claim 5 wherein the photoresist composition is a chemically amplified positive-acting photoresist composition.

(new) Claim 22. The method of claim 18 wherein the photoresist composition is a chemically amplified positive-acting photoresist composition.

(new) Claim 23. The substrate of claim 1 wherein the photoresist composition is a chemically amplified positive-acting photoresist composition.

(new) Claim 24. A method for forming a relief image on a substrate comprising:
applying on the substrate a layer of an antihalation composition comprising an anthracene material;
applying over the antihalation composition coating layer a photoresist composition; and
exposing the applied photoresist composition to patterned radiation having a wavelength of 248 nm.

(new) Claim 25. The method of claim 25 wherein the antihalation composition is crosslinked prior to applying the photoresist composition over the antihalation composition layer.

JUL-19-2007 THU 05:21 PM E A P & D

FAX NO. 6174394170

P. 08

Application No. 90/008,359

5

Docket No.: 40678-6C

(new) Claim 26. The method of claim 24 wherein the photoresist composition is a chemically-amplified positive-acting photoresist composition.

(new) Claim 27. The method of claim 25 wherein the photoresist composition is a chemically-amplified positive-acting photoresist composition.

(new) Claim 28. A method for forming a relief image on a substrate comprising:
applying on the substrate a layer of an antihalation composition comprising an anthracene material;
crosslinking the antihalation composition layer; and
applying over the crosslinked antihalation composition coating layer a photoresist composition.

(new) Claim 29. The method of claim 28 further comprising imaging the photoresist composition with activating radiation and treating the imaged photoresist composition with a developer to provide a photoresist relief image.

(new) Claim 30. The method of claim 29 wherein areas bared of photoresist upon treatment with the developer are etched.

(new) Claim 31. The method of claim 29 wherein areas bared of photoresist upon treatment with the developer are exposed to a plasma gas.

JUL-19-2007 THU 05:21 PM E A P & D

FAX NO. 6174394170

P. 09

Application No. 90/008,359

6

Docket No.: 40678-5C

REMARKS

Claims 1-31 are pending herein. Claim 28 has been amended to add "crosslinked" antihalation layer and thus recites "applying over the crosslinked antihalation composition coating layer a photoresist composition." No new matter has been added by that amendment. For instance, support for the amendment appears e.g. in the original claims of the application.

Patent Owner's Statement of Interview:

The undersigned and Dr. Thackeray appreciate the time and helpful comments provided by Examiners Stein and Jones during the interview on July 12, 2007. At that time, the amendment of claim 28 as made herein was discussed to provide further clarity. The Rhode document also was discussed. The Examiners agreed that U.S. Patent 4,863,827 (Jain) and U.S. Patent 3,884,702 (Koshimo) do not disclose anthracene materials.

Supplemental Response:

Claims 1-5 and 11-14 were rejected under 35 U.S.C. 102 over Rhode (U.S. Patent 4,935,320). The rejection is traversed.

Claims 1 and 5 (the only rejected independent claims) each calls for a "positive-acting photoresist."

The Rhode document does not describe use of positive photoresists. Rather, the Rhode document reports use of a negative composition only. See, for instance, column 32 of Rhode.

Indeed, it has been recognized that polyimide compositions are more typically employed as negative-acting compositions. Enclosed is a copy of an article "HD Micro Puts Out Positive Polyimide," Electronic News (June 19, 2000), which states "Virtually all the polyimides (before now) have been negative tone polyimides"

JUL-19-2007 THU 05:22 PM E A P & D

FAX NO. 6174394170

P. 10

Application No. 90/008,359

7

Docket No.: 40678-5C

The Rhode document also does not teach or suggest subject matters of the new claims.

For instance, new independent claim 24 recites "exposing the applied photoresist composition to patterned radiation having a wavelength of 248 nm." The Rhode document reports higher exposure wavelengths. See Rhode at column 2, lines 50-53.

New independent claim 28 recites "crosslinking the antihalation composition layer; and applying over the crosslinked antihalation composition coating layer a photoresist composition." Such crosslinking and application of a photoresist composition is not disclosed in Rhode.

Accordingly, the rejection is properly withdrawn. See *In re Marshall*, 198 USPQ 344, 346 (CCPA 1978) ("[r]ejections under 35 U.S.C. §102 are proper only when the claimed subject matter is identically disclosed or described in the prior art.").

It is believed the application is in condition for immediate allowance, which action is earnestly solicited.

Respectfully submitted,

By 

Peter F. Corless

Registration No.: 33,860

EDWARDS ANGELL PALMER & DODGE
LLP

P.O. Box 55874

Boston, Massachusetts 02205

(617) 439-4444

Attorneys/Agents For Applicant

JUL-19-2007 THU 05:22 PM E A P & D

FAX NO. 6174394170

P. 11

HD Micro Puts Out Positive Polyimide

-- 6/19/2000

Electronic News

HD MicroSystems LLC said it has developed a positive photodefinable polyimide that will save chipmakers process costs and mask steps.

HD Micro, a joint venture between DuPont Co. and Hitachi Chemical Co. Ltd., introduced its HD-8000 polyimide developed for single mask, stress buffer passivation applications such as DRAMs, SRAMs, logic devices and microprocessors. The polyimide layer is applied to a wafer on top of the final passivation layer in the last production step before back grinding, dicing and packaging.

"All of the DRAM manufacturers use the overcoat technology for protection," noted Ian Matthews, a chief scientist for HD Micro. About half of logic device manufacturers currently utilize a polyimide layer, he said.

"Virtually all of the polyimides (before now) have been negative tone polyimides," Matthews said. Since chipmakers moved to positive, as opposed to negative, resists in the 1980s, this has meant an extra set of mask steps is necessary to apply a final polyimide layer to a wafer. "With DH-8000, they can use the masks they've already created," Matthews said.

"The negative tone systems out there require solvent developers, and that requires a special, additional track," added Craig Schuckert, global product manager for HD Micro. With the positive tone HD-8000, which doesn't contain NMP, a chemical commonly found in polyimides that is not compatible with deep-ultraviolet resists, maintaining a separate track and the associated disposal steps are not longer necessary, Schuckert said.

HD-8000 can be processed in fabs using both deep-ultraviolet and I-line photoresists, according to HD Micro. The company said that with an aspect ratio of two to one, HD-8000 is capable of imaging 4 micron features in 8 micron cured films, making it ideal for use as a silicon nitride etch dry mask. HD-8000 has a cure cycle of 90 minutes, a glass transition temperature of 300 degrees Celsius and a cured film thickness range of 5 to 10 microns. It can be patterned with both I-line and G-line steppers. HD Micro expects to begin shipment of the new polyimide in the third quarter of this year.

© 2007, Reed Business Information, a division of Reed Elsevier Inc. All Rights Reserved.

JUL-19-2007 THU 05:22 PM E A P & D

FAX NO. 6174394170

P. 12

In re Application:
Thackeray et al.

Application No.: 90/008,359

Confirmation No.: 8757

Filed: December 4, 2006

Art Unit: 3991

For: ANTIHALATION COMPOSITIONS

Examiner: S. Stein

CERTIFICATE OF SERVICE

I, Peter F. Corless hereby certify that in connection with the above-identified application copies of the following filed with the U.S. Patent and Trademark Office on July 19, 2007:

Supplemental Amendment
Amendment Transmittal Letter

are being deposited with the United States Postal Service on July 19, 2007, in an envelope with first class postage addressed to:

Constance S. Huttner
Skadden Arps Slate Meagher & Flom LLP
Four Times Square
New York, New York 10036



Peter F. Corless (Reg. 33860)
EDWARDS, ANGELL, PALMER & DODGE LLP
P.O. Box 55874
Boston, MA 02205
(617) 439-4444

EXHIBIT DD

Chapter 600 Parts, Form, and Content of Application

| | | | |
|------------|---|------------|---|
| 601 | Content of Provisional and Nonprovisional Applications | 605.04(b) | One Full Given Name Required |
| 601.01 | Complete Application | 605.04(c) | Inventor Changes Name |
| 601.01(a) | Nonprovisional Applications Filed Under 35 U.S.C. 111(a) | 605.04(d) | Applicant Unable to Write |
| 601.01(b) | Provisional Applications Filed Under 35 U.S.C. 111(b) | 605.04(e) | May Use Title With Signature |
| 601.01(c) | Conversion to or from a Provisional Application | 605.04(f) | Signature on Joint Applications - Order of Names |
| 601.01(d) | Application Filed Without All Pages of Specification | 605.04(g) | Correction of Inventorship |
| 601.01(e) | Nonprovisional Application Filed Without At Least One Claim | 605.05 | Administrator, Executor, or Other Legal Representative |
| 601.01(f) | Applications Filed Without Drawings | 605.07 | Joint Inventors |
| 601.01(g) | Applications Filed Without All Figures of Drawings | 606 | Title of Invention |
| 601.01(h) | Forms | 606.01 | Examiner May Require Change in Title |
| 601.02 | Power of Attorney | 607 | Filing Fee |
| 601.03 | Change of Correspondence Address | 607.02 | Returnability of Fees |
| 601.04 | National Stage Requirements of the United States as a Designated Office | 608 | Disclosure |
| 601.05 | Bibliographic Information — Application Data Sheet (ADS) | 608.01 | Specification |
| 602 | Original Oath or Declaration | 608.01(a) | Arrangement of Application |
| 602.01 | Oath Cannot Be Amended | 608.01(b) | Abstract of the Disclosure |
| 602.02 | New Oath or Substitute for Original | 608.01(c) | Background of the Invention |
| 602.03 | Defective Oath or Declaration | 608.01(d) | Brief Summary of Invention |
| 602.04 | Foreign Executed Oath | 608.01(e) | Reservation Clauses Not Permitted |
| 602.04(a) | Foreign Executed Oath Is Ribboned to Other Application Papers | 608.01(f) | Brief Description of Drawings |
| 602.05 | Oath or Declaration — Date of Execution | 608.01(g) | Detailed Description of Invention |
| 602.05(a) | Oath or Declaration in Continuation and Divisional Applications | 608.01(h) | Mode of Operation of Invention |
| 602.06 | Non-English Oath or Declaration | 608.01(i) | Claims |
| 602.07 | Oath or Declaration Filed in United States as a Designated Office | 608.01(j) | Numbering of Claims |
| 603 | Supplemental Oath or Declaration | 608.01(k) | Statutory Requirement of Claims |
| 603.01 | Supplemental Oath or Declaration Filed After Allowance | 608.01(l) | Original Claims |
| 604 | Administration or Execution of Oath | 608.01(m) | Form of Claims |
| 604.01 | Seal | 608.01(n) | Dependent Claims |
| 604.02 | Venue | 608.01(o) | Basis for Claim Terminology in Description |
| 604.03(a) | Notarial Powers of Some Military Officers | 608.01(p) | Completeness |
| 604.04 | Consul | 608.01(q) | Substitute or Rewritten Specification |
| 604.04(a) | Consul — Omission of Certificate | 608.01(r) | Derogatory Remarks About Prior Art in Specification |
| 604.06 | By Attorney in Application | 608.01(s) | Restoration of Canceled Matter |
| 605 | Applicant | 608.01(t) | Use in Subsequent Application |
| 605.01 | Applicant's Citizenship | 608.01(u) | Use of Formerly Filed Incomplete Application |
| 605.02 | Applicant's Residence | 608.01(v) | Trademarks and Names Used in Trade |
| 605.03 | Applicant's Mailing or Post Office Address | 608.02 | Drawing |
| 605.04(a) | Applicant's Signature and Name | 608.02(a) | New Drawing — When Replacement is Required Before Examination |
| | | 608.02(b) | Informal Drawings |
| | | 608.02(c) | Drawing Print Kept in File Wrapper |
| | | 608.02(d) | Complete Illustration in Drawings |
| | | 608.02(e) | Examiner Determines Completeness and Consistency of Drawings |
| | | 608.02(f) | Modifications in Drawings |
| | | 608.02(g) | Illustration of Prior Art |
| | | 608.02(h) | Replacement Drawings |
| | | 608.02(i) | Transfer of Drawings From Prior Applications |
| | | 608.02(m) | Drawing Prints |

PARTS, FORM, AND CONTENT OF APPLICATION

601.03

601.03 Change of Correspondence Address [R-5]

37 CFR 1.33. Correspondence respecting patent applications, reexamination proceedings, and other proceedings.

****>**

(a) *Correspondence address and daytime telephone number.* When filing an application, a correspondence address must be set forth in either an application data sheet (§ 1.76), or elsewhere, in a clearly identifiable manner, in any paper submitted with an application filing. If no correspondence address is specified, the Office may treat the mailing address of the first named inventor (if provided, see §§ 1.76(b)(1) and 1.63(c)(2)) as the correspondence address. The Office will direct all notices, official letters, and other communications relating to the application to the correspondence address. The Office will not engage in double correspondence with an applicant and a patent practitioner, or with more than one patent practitioner except as deemed necessary by the Director. If more than one correspondence address is specified in a single document, the Office will select one of the specified addresses for use as the correspondence address and, if given, will select the address associated with a Customer Number over a typed correspondence address. For the party to whom correspondence is to be addressed, a daytime telephone number should be supplied in a clearly identifiable manner and may be changed by any party who may change the correspondence address. The correspondence address may be changed as follows:

(1) *Prior to filing of § 1.63 oath or declaration by any of the inventors.* If a § 1.63 oath or declaration has not been filed by any of the inventors, the correspondence address may be changed by the party who filed the application. If the application was filed by a patent practitioner, any other patent practitioner named in the transmittal papers may also change the correspondence address. Thus, the inventor(s), any patent practitioner named in the transmittal papers accompanying the original application, or a party that will be the assignee who filed the application, may change the correspondence address in that application under this paragraph.<

(2) *Where a § 1.63 oath or declaration has been filed by any of the inventors.* If a § 1.63 oath or declaration has been filed, or is filed concurrent with the filing of an application, by any of the inventors, the correspondence address may be changed by the parties set forth in paragraph (b) of this section, except for paragraph (b)(2).

37 CFR 1.33(a) provides that the application must specify a correspondence address to which the Office will send notice, letters, and other communications relating to an application. The correspondence address must either be in an application data sheet (37 CFR 1.76) or in a clearly identifiable manner elsewhere in any papers submitted with the application filing. If more than one correspondence address is specified in a single document, the Office will

****>**select one of the specified addresses for use as the correspondence address and, if given, will select the address associated with a Customer Number over a typed correspondence address.< Additionally, applicants will often specify the correspondence address in more than one paper that is filed with an application, and the address given in the different places sometimes conflicts. Where the applicant specifically directs the Office to use non-matching correspondence addresses in more than one paper, priority will be accorded to the correspondence address specified in the following order: (A) Application data sheet (ADS); (B) application transmittal; (C) oath or declaration (unless power of attorney is more current); and (D) power of attorney. Accordingly, if the ADS includes a typed correspondence address, and the declaration gives a different address (i.e., the address associated with a Customer Number) as the correspondence address, the Office will use the typed correspondence address as included on the ADS. In the experience of the Office, the ADS is the most recently created document and tends to have the most current address. After the correspondence address has been entered according to the above procedure, it will only be changed pursuant to 37 CFR 1.33(a)(1).

The submission of a daytime telephone number of the party to whom correspondence is to be addressed is requested pursuant to 37 CFR 1.33(a). While business is to be conducted on the written record (37 CFR 1.2), a daytime telephone number would be useful in initiating contact that could later be reduced to writing. Any party who could change the correspondence address could also change the telephone number.

37 CFR 1.33(a)(1) provides that the party filing the application and setting forth a correspondence address may later change the correspondence address provided that an executed oath or declaration under 37 CFR 1.63 by any of the inventors has not been filed. If a ****>**patent practitioner (i.e., registered attorney or agent)< filed the application, any other ***>**patent< practitioners named in the transmittal letter may **>also<** change the correspondence address. A ***>**patent< practitioner named in a letterhead would not be considered as being named in the transmittal letter for purposes of changing the correspondence address. A clear identification of the individual as a representative would be required. If an application is filed by a company to whom the invention has been

assigned or to whom there is an obligation to assign the invention, a person who has the authority to act on behalf of the company may change the correspondence address. Thus, the inventor(s), any *>patent<* practitioner named in the transmittal papers accompanying the original application, or a party that will be the assignee who filed the application, may change the correspondence address pursuant to 37 CFR 1.33(a)(1). The filing of an executed oath or declaration that does not include a correspondence address does not affect any correspondence address previously established on filing of the application, or changed pursuant to 37 CFR 1.33(a)(1).

Where a correspondence address has been established on filing of the application or changed pursuant to 37 CFR 1.33(a)(1) (prior to the filing of an executed oath or declaration under 37 CFR 1.63 by any of the inventors), that correspondence address remains in effect upon filing of an executed oath or declaration under 37 CFR 1.63 and can only be subsequently changed pursuant to 37 CFR 1.33(a)(2). Under 37 CFR 1.33(a)(2), where an executed oath or declaration under 37 CFR 1.63 has been filed by any of the inventors, the correspondence address may be changed by (A) a *>patent practitioner<* of record, (B) an assignee as provided for under 37 CFR *>3.71<*(b), or (C) all of the applicants (37 CFR 1.41(b)) for patent, unless there is an assignee of the entire interest and such assignee has taken action in the application in accordance with 37 CFR 3.71. See 37 CFR 1.33(a)(2).

Where an attorney or agent of record (or applicant, if he or she is prosecuting the application *pro se*) changes his or her correspondence address, he or she is responsible for promptly notifying the U.S. Patent and Trademark Office of the new correspondence address (including ZIP Code). *>See 37 CFR 11.11.<* The notification should also include his or her telephone number. A change of correspondence address may not be signed by an attorney or agent not of record (see MPEP § 405).

Unless the correspondence address is designated as the address associated with a Customer Number, a separate notification must be filed in each application for which a person is intended to receive communications from the Office. See MPEP § 403 for Customer Number Practice. In those instances where a change

in the correspondence address of a registered attorney or agent is necessary in a plurality of applications, the notification filed in each application may be a reproduction of a properly executed, original notification. The original notice may either be sent to the Office of Enrollment and Discipline as notification to the Attorney's Roster of the change of address, or may be retained by applicant. See MPEP § 502.02.

Special care should be taken in continuation or divisional applications to ensure that any change of correspondence address in a prior application is reflected in the continuation or divisional application. For example, where a copy of the oath or declaration from the prior application is submitted for a continuation or divisional application filed under 37 CFR 1.53(b) and the copy of the oath or declaration from the prior application designates an old correspondence address, the Office may not recognize, in the continuation or divisional application, the change of correspondence address made during the prosecution of the prior application. Applicant is required to identify the change of correspondence address in the continuation or divisional application to ensure that communications from the Office are mailed to the current correspondence address. 37 CFR 1.63(d)(4).

See MPEP § 711.03(c) for treatment of petitions to revive applications abandoned as a consequence of failure to timely receive an Office action addressed to the old correspondence address.

The required notification of change of correspondence address need take no particular form. However, it should be provided in a manner calling attention to the fact that a change of address is being made. Thus, the mere inclusion, in a paper being filed for another purpose, of an address which is different from the previously provided correspondence address, without mention of the fact that an address change is being made would not ordinarily be recognized or deemed as instructions to change the correspondence address on the file record.

The obligation (see 37 CFR 11.11) of a registered attorney or agent to notify the Attorney's Roster by letter of any change of his or her address for entry on the register is separate from the obligation to file a notice of change of address filed in individual applications. See MPEP § 402.